(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization

International Bureau



(43) International Publication Date 17 March 2005 (17.03.2005)

(10) International Publication Number WO 2005/024745 A2

(51) International Patent Classification7:

G08B

(21) International Application Number:

PCT/GB2004/003821

(22) International Filing Date:

8 September 2004 (08.09.2004)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

10/657,283 8 September 2003 (08.09.2003) 10/734,813 12 December 2003 (12.12.2003) US 10/779,517 13 February 2004 (13.02.2004) US 10/920,094 17 August 2004 (17.08.2004) US

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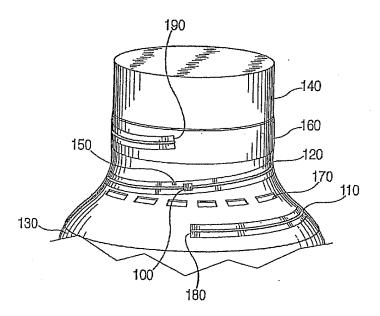
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- (81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NA, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RU, SC, SD, SE, SG, SK, SL, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM,
- (84) Designated States (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

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(54) Title: BOTTLE CAP



(57) Abstract: An apparatus and method for detecting tampering with a container sealed with a cap, comprising a radio frequency identification (RFID) tag mounted on the container. When the container is tempered with the tag is disabled and this is detected. The apparatus may also comprise a tag system for use with a container having a metal closure, the system being mounted internally or externally of the metal closure. The apparatus may also be adapted to verify the payment of taxes using an RFID tag mounted on the container.

WO 2005/024745 A2



Published:

 without international search report and to be republished upon receipt of that report For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

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BOTTLE CAP

FIELD OF THE INVENTION

The invention relates generally to bottle caps and to seals for containers. In particular it relates to an apparatus and method that detects tampering with containers that are sealed with caps, such as bottles, and prevents counterfeiting thereof. More specifically, in a first embodiment, an RFID tag attached to a bottle is disabled upon tampering with the bottle. In a second embodiment, a sensor is integrated with a radio frequency identification ("RFID") tag such that the output of the RFID tag provides an indication of whether the bottle has been tampered with, as determined by the sensor. In another embodiment it relates to an apparatus and method for providing an RFID tag on a metal closure for a container such as a metal bottle cap.

The invention further relates generally to an apparatus that electronically determines whether a tax for a product has been paid. In addition, it can verify the authenticity of the contents within a container closed with a cap or other closure, such as a bottle, and automatically disable itself when first opened.

BACKGROUND OF THE INVENTION

The wine and spirits industry loses significant revenue due to illicit activities in the supply chain. One of the problems is a proliferation of counterfeit goods. Another problem is goods that have been tampered with, where a tamperer will drain them of their original contents and replace them with a cheap and inferior substitute. The concern with such illicit activities goes even beyond the loss of the sale itself because the proliferation of a poor quality counterfeit product may hurt the reputation of an otherwise well respected label. Furthermore, there are concerns that a counterfeit product or one that has been tampered with may be contaminated with substances that could cause serious health problems. The industry has tried to tackle this illicit activity by implementing countermeasures such as holograms to help identify a

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genuine product. However these efforts have been largely unsuccessful because counterfeiters have the capability of reproducing them. Consequently, counterfeit goods are virtually indistinguishable from the legitimate goods without directly examining the contents of such goods, which requires actually opening the packaging of such goods. In the case of most goods, and especially beverages and other foodstuffs, opening the packaging renders the goods worthless since they can not be resold. Therefore, there is a need for a secure system that will allow anyone in the supply chain, from the manufacturer to the customer, to easily detect whether a container has been tampered with or is counterfeit.

It is therefore an object of the present invention to provide an improved apparatus and method for detecting tampering with containers which can not be easily duplicated by counterfeiters.

It is a further object of the present invention to provide an improved apparatus and method for detecting tampering with containers with allows anyone in the supply chain to detect whether a container has been tampered with or is counterfeit.

However, the use of an RFID tag with a metal container closure or cap present certain design difficulties when compared to mounting an RFID tag within a plastic cap for a container, e.g., a beverage bottle. The plastic material does not significantly affect the transmission of the electromagnetic signal transmitted to the RFID tag. As used herein, metal cap is understood to mean any metal closure for any type of container. Furthermore, references herein to bottles and metal caps for bottles is not to be understood as limiting the scope of the invention but merely illustrative of a particular application for the invention. At the high RF frequencies used for communication with an RFID tag, some transmitted signal energy will diffract and reflect into a metal cap from the open end of the metal cap so long as the fluid contents within the container remain below the bottom of the cap. However, a full container will

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likely prevent the RF signal from reaching an RFID tag mounted within a metal cap. Furthermore, since an RFID tag normally does not include an integral battery and is powered by the received RF energy, sufficient RF energy has to reach the RFID tag to power the integrated circuit chip on the RFID tag. It is unlikely that this would occur for an RFID tag mounted within a metal cap absent special circumstances, such as positioning the interrogator antenna at a very close range and at a specific orientation to the metal cap. Consequently, a conventional RFID tag mounted completely inside a metal cap does not appear to be practical.

Microstrip antenna technology originated in microwave transmission lines etched into radio frequency integrated circuits and into copper-clad printed circuit boards. A microstrip transmission line is a metal conductor path (usually etched copper) separated from an expansive conducting surface (ground plane) by an insulating dielectric layer. The width of the transmission line and the thickness of the dielectric medium determine the characteristic impedance of the transmission line, and thereby the efficiency of RF power transmission from one device to another. If the length of the microstrip transmission line is adjusted to be one-half the wavelength of RF waves in the dielectric layer, and if one or both ends of the transmission line are not connected to a device, then that transmission line radiates energy (or receives it) as an antenna. Consequently, the same technology and the same process steps can be used to produce an antenna and the necessary impedance matching components, resulting in lower manufacturing costs.

For these reasons, microstrip antennas are commonly used in connection with the interrogator of a RFID system. These antennas have the desirable characteristic of laying flat on a surface with minimum protrusion from that surface. However, they are not commonly used on RFID tags, primarily for the following three reasons: 1) The characteristic length of a simple microstrip antenna is one-half of the wavelength, whereas it is one-quarter of the

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wavelength for an electric dipole antenna. Consequently, for a given frequency of operation, the microstrip antenna must be twice the length the electric dipole antenna. 2) The simplest microstrip antennas have a narrower bandwidth than the electric dipole antenna, resulting in tighter manufacturing tolerances for the microstrip antenna. 3) Since the patch of the microstrip antenna is more massive than the wire antenna, the RFID tag IC chip must have more substantial power conversion and switching devices than is necessary for the wire antenna in order to modulate the backscattered RF energy return to the interrogator.

The use of a microstrip antenna for an RFID tag has been disclosed in U.S. Patent No. 6,215,402, which includes several designs for patch antennas and impedance matching components for an RFID tag, and U.S. Patent No. 6,329,915, which describes the use of an additional insulating material with high electric permittivity that is applied to the surface on top of the microstrip antenna in order to further reduce the size of the antenna. However, neither of these patents discloses the use of an RFID tag having a microstrip antenna on a metal closure for a container.

The use of specially designed slots etched into the interior of a patch antenna to broaden the bandwidth of a microstrip antenna without changing the overall form factor of the antenna is disclosed in an article by Ali, Sittironnarit, Hwang, Sadler, and Hayes, entitled "Wideband/Dual-Band Packaged Antenna for 5-6 GHz WLAN Application," that appeared in the February, 2004 issue of the journal IEEE Transactions on Antennas and Propagation. However, this article does not disclose the use of an RFID tag having a microstrip antenna on a metal bottle cap.

Accordingly, it is another object of the present invention to provide an RFID tag employing an antenna that can be mounted on the exterior of a metal closure for a container and that provides the same functionality as a conventional RFID tag mounted on a plastic closure for a container.

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It is a further object of the present invention to provide an RFID tag for mounting on a metal cap that is not subject to close tolerances in manufacturing.

Another problem associated with the sale of the alcoholic beverages is insuring that the tax or duty has been paid each bottle sold. It has been estimated that many millions of dollars of governmental revenues are lost each year because required taxes are not paid. One solution currently under discussion in the United Kingdom is to have stamps, in the form of strips of paper, placed over the top of the bottle's package to show that the tax has been paid for that package.

It is anticipated that radio frequency identification ("RFID") devices (commonly called "RFID tags") will soon replace bar codes as the chief way to identify goods. An RFID tag is pre-encoded to include information about the associated product to which it is attached or to contain a distinctive identification code. When an RFID tag is interrogated by an external reader, it will provide a radio signal containing that pre-encoded data. Thus, for example, a code can be encoded within the RFID tag that defines the product during checkout and this information can be read (instead of a bar code) to determine the type of goods and calculate its price (which is either also encoded in the RFID tag or stored in a main database and obtained based upon the product code). However, a great deal of concern has arisen recently relative to privacy issues because conventional RFID tags continue to be active long after a product has been purchased. This means that third parties can continue to read the information in the RFID tag for the life of the product, for example, allowing these third parties to generate information about a person's shopping tendencies without their knowledge.

As a result, although RFID tags can be used to distinguish between legitimate goods and counterfeit goods because the counterfeit goods will not include valid RFID tags because of the difficulty in reproducing RFID tags, the

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use of such RFID tags on goods is likely to raise privacy concerns if the RFID tag remains active for the life of the product.

It is therefore another object of the present invention to provide an RFID tag for an alcoholic beverage package which can be programmed to verify that any applicable tax or duty for that package has been paid.

It is yet another object of the present invention to provide an improved apparatus and method for verifying the authenticity of the contents of a container which can not be easily duplicated by counterfeiters and which is permanently disabled upon the opening of the container for the first time.

It is a further object of the present invention to provide an improved apparatus and method for verifying the authenticity of the contents of a container which can also be permanently disabled electronically.

SUMMARY OF THE INVENTION

The first aspect of the present invention is an apparatus and method for detecting tampering with a container sealed with a cap. Tampering is detected by disabling an RFID tag mounted on the container. Alternatively, the RFID tag transmits a first message when no tampering has been detected, and a second message after detecting tampering. Counterfeit items can also be detected, since only those containers including the apparatus of the present invention are deemed original.

Preferably, the apparatus includes a radio frequency transceiver circuit which will transmit a signal in response to receipt of a command, an antenna having a fixed length and connected to the radio frequency transceiver circuit, and means for preventing the radio frequency transceiver circuit from transmitting a command when the container has been tampered with.

In a first embodiment, the apparatus includes a substrate having a first portion, a second portion, and, preferably, a perforation in the substrate at some point in between the first portion and the second portion. The first portion of the substrate is permanently affixed to a portion of the container in a location

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proximal to the cap. The second portion of the substrate is permanently affixed to the cap. A radio frequency transceiver circuit is adapted to transmit an identification code upon receipt of a command to transmit and is mounted on the substrate. An antenna having a fixed length is connected to the radio frequency transceiver circuit and is mounted on the substrate. A portion of the antenna is preferably mounted on the substrate over the perforation. The removal of the cap from the container causes the antenna to separate, preventing the radio frequency transceiver from receiving any commands. Preferably, the perforation on the substrate is separated upon removal of the cap from the container, causing the antenna to separate. Preferably, the first embodiment may further comprise a security seal mounted over the substrate, the radio frequency transceiver circuit and the antenna.

In a second embodiment, the apparatus includes a cap adapted to seal the container when connected thereto. A radio frequency transceiver circuit is adapted to transmit an identification code upon receipt of a command to transmit and is mounted within the cap. An antenna having a fixed length is connected to the radio frequency transceiver circuit and is mounted to the cap. A sensor that produces an output indicative of an amount of the substance within the container is mounted within the cap. A means for disabling the radio frequency transceiver when the sensor indicates a change in the amount of the substance within the container is also mounted within the container. Preferably, the sensor of the second embodiment comprises one resistor having a predetermined resistance and a probe connected as a voltage divider that is excited by a battery having a fixed voltage. The probe is formed from a first conductive rod and a second conductive second rod mounted substantially parallel to each other and perpendicular to a horizontal axis of the cap such that the probe has a low resistance when the first conductive rod and the second conductive rod are within the substance in the container and a high resistance when not immersed in the substance in the container. Alternatively, in the

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second embodiment, a means for modifying the identification code in response to a command to transmit may be substituted for the means for disabling, such that an alternative message is sent after tampering is detected.

Preferably, the present invention is used within containers used in the wine and spirits industry, but, as one of skill in the art will readily recognize, the present invention has a broad application and can be used on any filled container.

A second aspect of the present invention is directed to an RFID tag system which communicates with a base station at a predetermined frequency for use with a container having a metal closure. The RFID tag system includes an antenna and insulator adapted to be mounted to an exterior surface of the metal closure and an RFID chip coupled to said antenna and adapted to be coupled to the metal closure. In a first embodiment, the RFID chip is mounted outside the metal closure. In a second embodiment, the RFID chip is mounted within the metal closure and connected to the antenna outside the metal closure through an electrical feedthrough connection in the metal closure.

In a third aspect of the present invention, an apparatus for use in verifying the payment of taxes or duties for the contents of a sealed container is provided. The apparatus comprises a radio frequency identification tag that includes a radio frequency transceiver circuit adapted to transmit a signal upon receipt of a transmit command and mounted upon a substrate. The radio frequency identification tag is mounted on the container and includes information indicative of the payment of a tax for the container.

In accordance with one presently preferred embodiment of this third aspect of the present invention, the apparatus comprises a radio frequency identification tag including information indicative that the tax for the contents of the container has been paid comprising a substrate, a radio frequency transceiver circuit adapted to transmit a signal upon receipt of a transmit command and mounted upon the substrate, an antenna having a fixed length

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mounted upon the substrate and connected to the radio frequency transceiver circuit, and a first interconnection means affixed to the substrate. The apparatus further contains a cap for the container having a cylindrical portion, a closed end of the cylindrical portion and an open end of the cylindrical portion, the radio frequency identification tag mounted on an inner surface of the cylindrical portion of the cap adjacent to the closed end such that the first interconnection means faces centrally inward. Finally, the apparatus contains a second interconnection means mounted on the container for engaging the first interconnection means when the cap is installed on the container such that removing the cap from the container will exert force on the substrate resulting in the fracture of the substrate thereby causing the radio frequency identification tag to become permanently disabled.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other objects and advantages of the invention, as well as the details of the illustrative embodiments, will be more fully understood by reference to the following portion of the specification, taken in conjunction with the accompanying drawings, in which:

- FIG. 1 is a side view of a first embodiment of an apparatus according to the first aspect of the present invention integrated into a plastic security seal around a container and a cap;
- FIG. 2 is a side view of the apparatus integrated into the cap and attached to a container according to the second embodiment;
- FIG. 3 is a top view of the electronic assembly in the second embodiment of the apparatus integrated into the cap according to the first aspect of the present invention;
- FIG. 4 is a block diagram of an electronic assembly in a second embodiment of the apparatus according to the first aspect of the present invention;
 - FIG. 5 is a schematic drawing of the electronic circuit of the apparatus

according to the second embodiment;

- FIG. 6 is a timing diagram of input and output signals of various components in the electronic circuit of FIG. 5 according to the second embodiment.
- FIG. 7A is a perspective exploded view, FIG. 7B is a perspective view and FIG. 7C is a cross-sectional view of the apparatus according to the third aspect of the present invention;
 - FIGS. 8A, 8B and 8C are front, top and side views, respectively, of the RFID tag of the third aspect of the present invention;
- FIGS. 9A and 9B are side and top views, respectively, of the bottle insert assembly of the third aspect of the present invention;
 - FIGS. 10A and 10B are side and top views of the central column portion of the bottle insert assembly of the third aspect of the present invention, FIGS. 10C and 10D are side and top views of the plain disc portion of the bottle insert assembly of the third aspect of the present invention and FIGS. 10E and 10F are side and top views, respectively, of the ratchet wheel portion of the bottle insert assembly of the third aspect of the present invention;
 - FIGS. 11A and 11B are bottom and side views of the bottle cap of the third aspect of the present invention;
- FIGS. 12A and 12B show an exploded view of the 2 parts of the bottle cap of the third aspect of the present invention.
 - FIG. 13 is a sequential series showing the assembly of the bottle cap of FIG. 12;
- FIG. 14 is an enlarged view of an embodiment of the top of the bottle insert assembly shown in FIG. 9a;
 - FIG. 15 is perspective view of a metal bottle cap including an RFID tag mounted on a top thereof according to one aspect of the present invention;
 - FIG. 16 is a plot of the length of a microstrip antenna versus the dielectric permittivity of the corresponding insulating layer that is used to

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calculate the size of the microstrip antenna for different applications according to another aspect of the present invention;

FIG. 17 is circuit diagram of a first embodiment of the second aspect of the present invention;

FIG. 18 is a circuit diagram of a second embodiment of the second aspect of the present invention; and

FIGS. 19A and 19B are circuit diagrams of a third embodiment of the second aspect of the present invention.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

The first aspect of the present invention relates to an apparatus for detecting counterfeit or altered containers. FIG. 1 shows a side view of a first embodiment of the apparatus according to the present invention. In this embodiment, a radio frequency identification ("RFID") chip 100 is connected to an antenna 110 having predetermined transmission characteristics controlled by the length thereof so that the RFID chip 100 may respond to radio frequency signals sent from another device known as an interrogator. Preferably, the RFID chip 100 is the so-called "mu-chip" manufactured and sold by Hitachi, but could also constitute any similar product sold by other manufacturers, including but not limited to products from Nippon Electric in Japan and Intermec in the U.S. The RFID chip 100 is a radio frequency back-scatter transceiver that transmits a stored identification code upon receipt of a command to transmit. Preferably, RFID chip 100 is passive, i.e. it uses the received signal to power itself. As one of skill in the art will readily recognize, a non-passive chip (i.e. an active chip) could be used for RFID chip 100 so long as a power source, e.g., a battery, is also included.

The RFID chip 100 and the antenna 110 are mounted on a substrate 120, preferably formed of polyimide. The substrate 120 includes a perforation 150 close to where RFID chip 100 is mounted and is permanently affixed to a container 130 on a first end 180 and to a cap 140 that has sealed the container

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130 on a second end 190. The substrate 120 is affixed in such a manner that the antenna 110 is wrapped around the circumference of the container 130 and the cap 20. When the cap 140 is twisted to remove it from the container 130 the polyimide substrate 120 ruptures at perforation 150. This causes a section of the antenna 110 to be separated and thus prevents the RFID chip 100 from transmitting or receiving signals from the interrogator, due to the change in the characteristics of antenna 110. In order to protect the RFID chip 100, the antenna 110 and the substrate 120 are preferably covered with a security seal 160 which has a multiplicity of perforations 170 along which it separates when the bottle cap 140 is removed from the bottle. The cap is preferably a plastic cap.

To verify the authenticity of the container 130, an interrogator is used to transmit an interrogation signal appropriate for the particular chip chosen for the RFID chip 100 (when the antenna 110 is connected and not separated). The interrogation signal may simply tell the RFID chip 100 to activate itself and send a response signal. Alternatively, the interrogation signal may be a more complex code or instruction that would require the RFID chip 100 to send a particular message in the response signal. Additionally, the response signal may contain information about the contents in the container such as the amount and type. The interrogator, however, will not receive a response signal if the container 130 has been opened, indicating it has been tampered with, or if the container 130 does not contain the apparatus at all, indicating that it is counterfeit, since RFID chip 100 will not receive the transmitted signal due to the change in characteristics of antenna 110. Therefore, in this first embodiment, a tampered bottle will be identified by its failure to respond to an interrogation.

FIGS. 2 and 3 depict the second embodiment of the present invention, and, in particular, show how electronic assembly 200 of FIGS. 4 and 5 is integrated into a cap 320 that seals a container 310.

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FIG. 2 shows the electronic assembly 200 of FIGS. 4 and 5 integrated into a cap 320 and attached to a container 310. Probe 205 is formed from two rods 340 and 350, which are preferably substantially parallel to each other and conductive -- preferably made of stainless steel. Rods 340 and 350 protrude from a point that is substantially in the center of the cap 320. An adhesive 360 is preferably placed around the probe 205, in order to form a liquid tight seal ensuring that the other portions of the electronic assembly 200 are protected from contamination. The cap 320 seals the container 310 filled with contents 330, and, when sealed, the bottom portions of the rods 340 and 350 extend into the contents 330. The two rods 340 and 350 are sized to a length so that their end portions remain in contact with the contents 330 regardless of the angle at which the container 310 is oriented.

In FIG. 3, block 300 represents battery 245, logic chip 225, Q-spoiler antenna 262 and voltage divider 240 of FIG. 4. The components within block 300 and RFID chip 235 are encased within the cap 320 and the two portions of antenna 230 go around the circumference of the cap 320. As one of skill in the art will readily recognize, depending upon the size of the cap 320, the two antenna portions 230 may run along the inner circumference or the outer circumference of the cap 320, or may even be embedded within the walls of the cap 320.

FIG. 4 is a block diagram of the electronic assembly 200. The electronic assembly 200 is comprised of a probe 205, resistor 210, a logic chip 225, an RFID chip 235, a two-part antenna 230, a Q-spoiler antenna and a battery 245.

Resistor 210 combines with probe 205 to form a voltage divider 240. Preferably, resistor 210 has a resistance of 10 Mohm, but as one of skill in the art will readily recognize, this value depends on the resistance of probe 205. As shown in FIG. 2, voltage divider 240 includes three nodes, node 290, 295 and 296. As one of skill will readily recognize, when a fixed voltage is applied between nodes 290 and 295, a signal will be obtained between nodes 296 and

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295 that will vary as the resistance of probe 205 changes.

Q-spoiler antenna 262 is an electrically conductive body of arbitrary size, shape and spatial orientation such that when it is electrically connected to antenna 230, by logic chip 225, the resonant property of antenna 230 is effectively diminished. In many cases, the Q-spoiler antenna 262 may be the negative electrode of battery 245.

Battery 245 has a positive terminal 250 and a negative terminal 255 and is used to excite the voltage divider 240 and to power logic chip 225, and is preferably of the lithium coin package style. Positive terminal 250 of battery 245 is connected to positive power connection 260 of the logic chip 225 and to node 290 of the voltage divider 240. The negative terminal 255 of battery 245 is connected to the negative power connection 265 (ground) to node 295 of the voltage divider 240, and to node 263 of the Q-spoiler antenna 262.

The logic chip 225, which is discussed in more detail with respect to FIG. 5, includes a positive input terminal 280 that is connected to a node 296 of the voltage divider 240. The logic chip 225 also has a first output terminal 270 and a second output terminal 275 that are connected to the antenna 230 at nodes 232 and 234, respectively. The RFID chip 235 also has a first output terminal 236 and a second output terminal 238 that are connected to the antenna 230 at node 232 and node 234, respectively. The voltage divider 240 provides a signal between nodes 296 and 295 that varies with the resistance of probe 205. The logic chip 225 monitors the signal provided by voltage divider 240, and, in turn, can generate an output signal on output terminals 270, 275 that prevents the RFID chip 235 from responding to an interrogation signal, depending on the input signal to logic chip 225 provided by voltage divider 240. Thus, for example, when probe 205 is immersed within the contents of a container, the resistance of probe 205 will be much lower than the resistance of resistor 210, and the voltage signal provided by voltage divider between nodes 296 and 295 will be low. Logic chip 225 allows RFID chip 235 to operate

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normally when the signal input at terminals 280, 265 is low (for the first instance of insertion of probe 205 into the contents of a container). However, if the contents of the container are emptied (or if the probe 205 is removed from the contents of the container), the resistance of probe 205 will greatly increase, generating a high voltage between nodes 296 and 295 (and thus at input terminals 280, 265), and causing logic chip 225 to disable RFID chip 235. If probe 205 is reinserted into the contents of the container, after having been removed from the container, logic chip 225 continues to disable RFID chip 235.

As discussed above, the voltage signal output between nodes 296 and 295 by the voltage divider 240 of FIG. 4 is a function of the resistance of probe 205, and thus is a function of the resistance between the rods 340 and 350. When the probe 205 is immersed in the contents 330 of the container 310, as shown in FIG. 4, the resistance between the rods 340 and 350 is of the order of 100 kohms and the voltage difference between nodes 296 and 295 of the voltage divider 240 will be low. However, when the probe 205 is removed from the contents 330, the resistance between the rods 340 and 350 is on the order of 100 Mohms, and the voltage difference between nodes 296 and 295 of the voltage divider 240 will be high. The voltage difference between nodes 296 and 295 of the voltage divider 240, therefore, provides an output signal that indicates whether the probe 205 is immersed in the contents 330.

As one of skill in the art will readily recognize, any mechanism that can detect the presence of a fluid (or other contents) in a container may be used instead of the voltage divider 240. For example, the level sensor may be a system that detects changes in the resonant frequency in the air space between the cap 320 and the contents 330. Other alternative level sensors include one that detects changes in the distance of a float that rests on top of the contents 330, or one that senses a change in air pressure within the container.

FIG. 5 is a schematic drawing that illustrates in detail the components

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comprising logic chip 225 of FIG. 4. In FIG. 5, all components common to FIG. 4 share the same reference number and operate in an identical manner. For convenience, battery 245 is shown with positive terminal 250 connected to +V (node 505) and the negative terminal 255 connected to common (node 500), and the power connections for logic chip 225 are not shown. In Fig. 5, Q-spoiler antenna 262 is shown connected to common (node 500).

The output of the voltage divider 240, i.e., the voltage between nodes 296 and 295, is provided to input terminal 297 of a series connection of resistor 215 and capacitor 220. The output terminal 298 of the series connection of resistor 215 and capacitor 220 is provided to input 527 of OR-gate 525, through input 280 of logic chip 225. The input port 527 of OR-gate 525 is a Schmitt trigger input, such as is constructed in the 74HC7002 specification of a 2-input OR gate. The combined effects of the series connection of resistor 215 and capacitor 220, together with the Schmitt trigger input of OR-gate 525 is a debounce circuit 222, as is commonly known to those of skill in the art. The output of de-bounce circuit 222 will be a logic high when probe 205 is not immersed in the contents of container 310 of FIG. 4, and will be a logic low when probe 205 is immersed in the contents of container 310. The Schmitt trigger output signal is clean and stable and essentially functions as a way to convert the noisy and naturally varying analog output signal of the voltage divider 240 into a digital signal.

The output signal of the OR-gate 525 is connected to the clock input 513 of D flip-flop 515. Input 513 is a positive-edge triggered clock input terminal of D flip-flop 515. The non-inverting output 511 of D flip-flop 515 is connected to a second input of OR-gate 525 (which may be a Schmitt trigger input, or may be a conventional input). The non-inverting output 511 of D flip-flop 515 also is connected to the single input terminals of first and second inverters 535 and 540 having open-drain outputs. A second output 519 of the D flip-flop 515 produces an inverted signal compared to the first output 511 and

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is fed back to a data input terminal 516 on the D flip-flop 515.

The output of the first and second inverters 535 and 540 are connected to the antenna 230 through the output terminals 270 and 275 of the logic chip 225. When their inputs are high, the low outputs of the first and second inverters 535 and 540 pull the outputs 236 and 238 of RFID chip 235 down, essentially shorting them to the Q-spoiling antenna 262, and preventing the RFID chip 235 from communicating with an interrogator.

D flip-flop 515 also includes a SET input 518 and a CLEAR input 517, both of which are asserted low. Thus, as shown in FIG. 5, the SET input 518 is tied to the positive terminal 250 of battery 245 (node 505) at input 512 and thus is inactive. In addition, an RC timing network comprising a resistor 580 and a capacitor 550 is connected between +V (node 505) and common (node 500). As one of skill in the art will readily recognize, the signal at node 514 will be low when power is first applied (e.g., when the battery 245 is installed) and will, at a rate determined by the RC time constant, charge up until a high level (i.e., +V) is reached. In the preferred embodiment, resistor 580 is 1 megohm and capacitor 550 is 1 microfarad. Thus, when the battery 245 is first installed, the input to D flip-flop 515 will be low, clearing the D flip-flop 515 of any prior states and setting the output 511 thereof low. Thereafter, the CLEAR input 517 will not change so long as the battery 245 is not removed.

FIG. 6 is a timing diagram that illustrates the operation of logic chip 225. The first trace 600 represents the voltage across probe 205. The second trace 610 is the voltage at input 280 of logic device 225. The third trace 620 is the signal at the output 511 of the D flip-flop 515. The fourth trace 625 is the signal at output lines 270 and 275 which connect logic chip 225 to antenna 230. The first significant event occurs when battery 245 is installed, which must occur with the probe 205 not positioned in a fluid or other container contents. As such, the output of OR-gate 525 will be high, as discussed above, and the output of D-flipflop 515 will be low because it has been reset on installation of

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the battery 245, as discussed above. This is shown in the first portion 630 of the timing diagram of FIG. 6.

At point 670 the second significant event occurs, i.e., the cap 320 is installed on a container 310, so that probe 205 is put in contact with the contents 330 of container 310. During the periods 630 and 640 thereafter, the apparatus is "set" -- meaning that the RFID chip 235 will be able to respond to interrogation by an RFID interrogator. In particular, at point 670, the input 527 to OR-gate 525 begins to go low, becoming a low logic level in one millisecond or less, as determined by the values of resistor 215 and capacitor 220. When the input 527 to OR-gate 525 becomes low, the output of OR-gate becomes low. Since this negative-going edge signal is presented to the positive-edge-triggered clock input 513 of D flip-flop 515, there is no signal change at the outputs 511 and 519 of D flip-flop 515. Since the D flip-flop 515 had been cleared during battery insertion, the RFID chip 235 continues to be able to respond to interrogation by an RFID interrogator.

The third significant event 680 is the first removal of cap 320 (and the corresponding removal of probe 205 from the contents 330 of container 310). The logic chip 225 at this point disables the RFID chip 235 from communicating with an interrogator. In particular, upon the removal of probe 205 from the contents 330 of container 310, the input 527 to OR-gate 525 begins to go high, becoming a high logic level in one millisecond or less, as determined by the values of resistor 215 and capacitor 220. When the input 527 to OR-gate 525 becomes high, the output of OR-gate also becomes high. Since this positive-going edge signal is presented to the positive-edge-triggered clock input 513 of D flip-flop 515, the output toggles high, and the outputs 270 and 275 of the two inventors 535 and 540 short the antenna 230 to the Q-spoiler antenna 262. This is shown during period 650 of FIG. 6.

The fourth significant event 690 is any subsequent insertion or withdrawal of cap 320 to or from the contents 330 of container 310. As can be

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seen from FIG. 6, and in particular during period 660, regardless of the input 527 to OR-gate 525, the output of OR-gate 525 will not change again since input 526 is held high by output 511 of D flip-flop 515, and the output of D flip-flop 515 will not change again because it can only be clocked by a negative-to-positive transition in the output of OR-gate 525, thereby preventing the apparatus from being reset. Thus, once the cap 320 is removed from container 310, the RFID chip 235 is permanently disabled (unless, as one of skill in the art will readily recognize, the battery is removed and reinstalled). As a result, the container 310 will only be verified as authentic and not tampered with when it responds to interrogation — which only occurs if cap 320 is not removed.

When interrogated, the RFID chip 235 may be programmed to respond with information identifying the particular container 310, the contents 330 of the container 310, the amount of contents 330 in the container 22 and/or any other information that may be useful.

In an alternative to the second embodiment, the RFID chip 235 may be combined with the logic chip 225 on a single integrated circuit. As one of skill in the art will readily recognize, in this situation it is possible to program two different codes into the RFID chip 235 that maybe transmitted when interrogated. In this situation, the apparatus includes logic that allows a first response to be generated to an interrogation prior to the cap 320 being removed from container 310, as above, and a second response generated after being removed from container 310. Furthermore, one of skill in the art will readily recognize that this added functionality may also be provided by modifying logic chip 225 accordingly.

The present invention offers a great flexibility in offering four independent features, in that the communication capabilities of each device may be controlled electronically (e.g., at the point of sale), mechanically (i.e., the tag is disabled once the cap is removed by separating two antenna portions),

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based upon liquid level (i.e., once the cap is removed or contents have been siphoned out) and lack of programmability (i.e., each tag has its information fixed upon manufacture).

The second aspect of the present invention relates to an apparatus and method for providing an RFID tag on a metal closure. Referring now to the drawing, and in particular to FIG. 15 thereof, therein illustrated is a metal cap 2100 having a RFID tag 2110 preferably employing a microstrip patch antenna (not shown) where the RFID tag 2110 is bonded to the top of metal cap 2100. The top surface 2120 of RFID tag 2110 can thereafter be decoratively printed in the same manner as conventional metal caps.

As discussed above, the IC chip of RFID tag 2110 may be located either outside the metal cap or inside the cap. Locating the chip outside the cap results in lower manufacturing costs since no feed-through connections are required. However, there may be functional incentives to locate the chip inside the cap, in which case one or more electrical feed-through connections are required to conduct signals from the antennal patch to the IC chip.

The microstrip patch antenna is naturally adapted to metal caps because the metal cap serves as the ground-plane for the antenna. The complementary metal surface (i.e., the patch) of the microstrip antenna is positioned on top of the metal cap with an insulating spacer between the two metal surfaces.

Two radio frequency bands are allocated by the Federal Communications Commission for RFID systems, 2.4 GHz and 5.8 GHz. Both of these frequency bands are used for other applications, including wireless telephones and wireless local area networks.

The characteristic dimension of the antenna that causes it to be tuned to a specific frequency (and the harmonics of that frequency) is larger for the simple patch antenna (one-half wavelength) than it is for a one-quarter wavelength electric dipole antenna, although more complex patch antennas can be fabricated that are the same characteristic length. Consequently, the

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simplest (and least costly) of 2.45 GHz patch antennas would barely fit on top of the smallest standard metal cap (1 1/8 inch diameter). There are other design options that could make it possible, from a technical standpoint, to use 2.45 GHz, although at a higher manufacturing cost. Alternatively, the 5.8 GHz microstrip antenna has a characteristic dimension of less than 1 inch and thus fits more easily on the top of conventional metal bottle caps.

When using a microstrip patch antenna, the RFID IC chip may be located either outside of the metal cap or within the metal cap. Locating the IC chip on the outside surface results in lower manufacturing cost, since feed-throughs are required to connect the antenna to the IC chip when the IC chip is mounted within the metal cap. Although a single feed-through could be used to connect the antenna to the IC chip, thereby reducing manufacturing costs, when two feed-throughs are employed, the length of the antenna patch can be reduced by 50%.

The microstrip antenna is preferred for a metal cap because, when properly designed, (1) it is more efficient receiving and re-radiating the resonant RF energy, (2) it offers a low profile on the bottle cap and (3) there is sufficient space on the top of the bottle cap to place the antenna if the system is operated at 2.45 GHz or at 5.8 GHz. Furthermore, the higher frequency 5.8 GHz microstrip antenna allows more design freedom and could lead to a lower-cost metal cap with integral RFID tag.

The characteristic length of the antenna patch, and the dielectric permittivity of the insulating layer, determine the frequencies at which the antenna may be used. Consequently, the diameter of the metal cap is the main consideration in selecting one of the two frequency bands that have been allocated by the Federal Communications Commission in the U.S. for use in RFID systems. The 2.45 GHz frequency band is widely used for RFID applications, while only a few systems have been developed for RFID at the higher 5.8 GHz frequency band. However, relevant radio technology at 5.8

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GHz has been developed extensively for other applications such as cordless telephones and wireless local area networks.

The characteristic length of the antenna patch is plotted as a function of the dielectric permittivity of the insulating layer at frequencies of 2.45 GHz (plot 2160) and 5.8 GHz (plot 2150) in FIG. 16. The thickness of the dielectric layer also has an effect on the characteristic length, at a given frequency, but the effect is much less than the permittivity. As seen from the plots, in order for the antenna patch to fit on the smallest standard size metal cap in the U.S. (i.e. a diameter of 1 1/8 inch shown as line 2170 in FIG. 16), the dielectric permittivity of the insulator for a 2.45 GHz antenna must be 5 or greater since only that portion of plot 2160 lies beneath line 2170. However, since plot 2150 lies entirely beneath line 2170, the patch will fit on the cap with any dielectric material for the 5.8 GHz antenna.

A table of the dielectric permittivity for various low-loss insulating materials manufactured by the Rogers Corp. is shown in Table I.

		Relative dielectric
Product (Rogers)	Composition	constant
RT/duroid 5880	PTFE glass fiber	2.2
RT/duroid 5870	PTFE glass fiber	2.33
ULTRALAM 2000	PTFE woven glass	2.5
RT/duroid 6002	PTFE ceramic	2.94
RO3003	PTFE ceramic	3
RO3203	PTFE ceramic reinforced woven glass	3.02
TMM 3	Hydrocarbon ceramic	3.27
RO4003C	Hydrocarbon ceramic	3.38
RO4350B	Hydrocarbon ceramic	3.48
RO4450B	Hydrocarbon ceramic prepreg	3.54

TMM 4	Hydrocarbon ceramic	4.5
TMM 6	Hydrocarbon ceramic	6
RT/duroid 6006	PTFE ceramic	6.15
RO3006	PTFE ceramic	6.15
TMM 10	Hydrocarbon ceramic	9.2
TMM 10i	Hydrocarbon ceramic	9.8
RT/duroid 6010LM	PTFE ceramic	10.2
RO3010	PTFE ceramic	10.2
RO3210	PTFE ceramic reinforced woven glass	10.2

TABLE I

The data from Fig. 16 and Table I demonstrates that several dielectric materials are available for a 2.45 GHz RFID microstrip antenna, e.g., TMM6 and RO3210. However, it is important to note that the antenna efficiency and therefore the sensitivity and range of the RFID tag, diminishes at higher values of permittivity (e.g., TMM6 is preferable over RO3210). This increases the need for precise impedance matching when employing an RFID tag operating at 2.45 GHz.

10 First Embodiment

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Figure 17 is a circuit diagram illustrating a first embodiment of this aspect of the present invention which is based upon a 5.8 GHz frequency band design. The RFID tag 2210 includes a fiberglass insulator 2206 having a relative permittivity 2.5 that is bonded to the top of metal cap 2100, an antenna 2201 that is mounted upon fiberglass insulator 2206, IC chip 2203, microstrip impedance-matching elements 2202 and 2205 which are also are mounted upon fiberglass insulator 2206 and which couple antenna 2201 to IC chip 2203, and microstrip ¼ -wave transformer 2204 that is also coupled to IC chip 2203 and which couples RF signals to the ground plane (i.e., the metal forming cap 2100) and eliminates the need for any direct electric connections between metal cap

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2100 and the RFID circuit mounted on insulator 2206. This form of coupling is well known among those of skill in the art of RF design. The configuration of this embodiment provides the lowest RFID tag cost and is generally limited to applications communicating via a 5.8 GHz link, since for many applications there will be insufficient room on the top of the metal cap for a 2.45 GHz patch together with impedance matching elements and IC chip. Design details for the microstrip impedance matching elements 2202 and 2205 are known to those of skill in the art, see, e.g., K. Chang, RF and Microwave Wireless Systems, Section 3.9 "Microstrip Patch Antennas", Wiley Interscience ISBN 0-471-35199-7 (2000) which is incorporated herein by reference. The number of quarter wavelength sections required, and their specific dimensions, are selected on the basis of the width of the patch, the thickness of the dielectric, and the permittivity of the dielectric.

Second Embodiment

Since the simplest patch atennas have only a 2% to 5% bandwidth, it may be desirable in terms of manufacturability to increase the bandwidth of a microstrip patch antenna to ensure that RFID tags are not tuned away from the frequency of the associated interrogator due to variations in component tolerances that arise in the manufacturing process. As one of skill in the art will readily recognize, an RFID tag having an increased bandwidth will still be able to communicate with an associated interrogator, even if the center frequency of the RFID tag varies from its intended value because of manufacturing tolerances, the influence of nearby dielectric materials or other factors. One method to increase the bandwidth of a patch antenna is disclosed in U.S. Patent Publication No. 2003/0222763, incorporated herein by reference. In that publication, a method is disclosed that increases the bandwidth of a patch antenna by 14% or more by etching slots in the patch antenna. An example, based on the methods disclosed in this publication is shown in FIG. 18 for an RFID tag system 2310 that uses a 5.8 GHz patch antenna.

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In particular, the RFID tag system 2310 includes the same components as the RFID tag system 2210 of FIG. 17 and discussed above. The only change is the addition of a slot 2401 in patch antenna 2201. Slot 2401 in antenna 2201 is asymmetrically shaped, and it is located off-center on the patch antenna 2201 which provides patch antenna 2201 with the effect of being two antennas that are closely spaced in frequency, thereby increasing the bandwidth thereof.

Third Embodiment

In some applications, it may be necessary to position the RFID IC chip be inside the metal cap. For example, it may be necessary to employ the RFID tags of the present invention in a larger system having interrogators that operate at a 2.8 GHz transmission frequency. In that case, since, as discussed above, the antenna patch could take up most of the area on the top of a metal cap, only the antenna patch could be positioned outside the metal cap and the antenna connected to the RFID chip is mounted inside the cap and connected to the external antenna via a feed-through connection, i.e., a wire connection that passes through the metal cap.

FIGS. 19A and 19B disclose an RFID tag system 2410 that operates at 2.8 GHz. FIG. 19A is a top view of cap 2100 and shows an insulator 2206 mounted on top of cap 2100, and circular antenna 2300 mounted on top of insulator 2206. Preferably, insulator 2206 is formed from Duroid 6006 (or comparable) dielectric material. Antenna 2300 is connected to the components located within cap 2100 via feedpoint 2301. As one of skill in the art will readily recognize, the location of feedpoint 2301 may be adjusted to optimize the impedance matching to the transmission line 2202 (FIG. 19B) on the inside of cap 2100. FIG. 19B shows a bottom view of cap 2100, showing feedpoint 2301 connecting to transmission line 2202, which, in turn, is connected to transmission line 2205. As in the previous embodiments, transmission line 2205 is thereafter connected to RFID IC chip 2203. As one of skill in the art will readily recognize, the transmission lines 2202 and 2205 are used to

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optimize the coupling of patch antenna 2300 to IC chip 2203. IC chip 2203 is connected to transmission line 2204 for coupling to the ground plane formed by metal cap 2100 via connection 2302. IC chip 2203 and transmission line components 2202, 2204 and 2205 are attached to a thin substrate 2420. The physical connections between the transmissions lines 2202 and 2204 and connections 2301 and 2302, respectively, may be wire bonds, as shown, or alternatively, substrate 2420 may be connected in other ways, e.g., sweat soldered or ultrasonically bonded to the connections 2301 and 2302, as understood by one of skill in the art.

If the bandwidth of the system illustrated in FIGS. 19A and 19B proves to be too narrow due to manufacturing tolerance problems, etc., a band widening slot can be etched in antenna 2300 in a manner similar to that described with respect to the second embodiment of the present invention shown in FIG. 18.

The third aspect of the present invention relates to an apparatus for verifying the payment of taxes or duties for the contents of a container, and is based upon the inclusion of an RFID tag associated with the container. The RFID tag contains an electronic code which contains information indicating whether a duty has been paid for the product associated with such RFID tag, e.g., a bottle of spirits. The RFID tag may also be programmed to include identification of the contents as well as other information relevant to supply chain operations. An interrogator is used to communicate with the RFID tag and may be a portable hand-held device or may be built into a computer, cash register, security exit, or the like. Whereas large counterfeiting operations in various countries are able to manufacture bottles, bottle caps, plastic bottle inserts, and labels that closely resemble authentic packaging, those same counterfeiting operations cannot afford the cost of developing the small, highly integrated radio chip. The RFID system may work at any of a variety of radio frequencies; however, the microwave frequency 2.45 Gigahertz is presently

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preferred because its smaller wavelength results in a shorter antenna and thus a smaller tag than lower frequency RFID tags. In addition, this microwave frequency has the advantage of being approved in most countries of the world for RFID applications. Furthermore, RFID devices that operate at 2.45 GHz are produced and marketed by several companies worldwide. Finally, the use of the spread-spectrum communications protocol and special code strategies for the information stored in the RFID tags effectively blocks counterfeiters from creating counterfeit or black-market RFID tags in order to avoid the payment of taxes or duties. Preferably, the RFID tag is mounted in the container cap (or other closure) and will be read by RFID interrogators used by legitimate organizers of the supply chain. When the cap is installed on the container there is an interconnection between the cap and the container such that when the cap is first removed from the container the RFID tag is disabled.

FIG. 7A is an exploded perspective view, FIG. 7B is a perspective view and FIG. 7C is a cross-sectional view of a presently preferred embodiment of the present invention. A bottle 1100 includes threads 1120 for securing a bottle cap over mouth 1110 thereof. According to this presently preferred embodiment of the invention, bottle 1100 is fitted with a tubular, preferably plastic, bottle insert 1200, that is tightly fitted into the mouth 1110 of bottle 1100. Bottle insert 1200 includes a ratchet wheel 1240. Thereafter, bottle 1100 is fitted with a cap 1300, also preferably of plastic, that screws onto threads 1120. Cap 1300 includes an RFID tag 1310 mounted near the closed end of cap 1300 such that it is longitudinally aligned with the ratchet wheel 1240 when cap 1300 is screwed tightly onto bottle 1100, with a cylindrical catch 1318 mounted on RFID tag 1310 interlocked with one of the plurality of ratchets on ratchet wheel 1240 (as specifically shown in FIG. 7B).

One embodiment of an RFID tag 1310 adapted for use in the present invention is shown in FIGS. 8A, 8B and 8C. As shown in FIG. 8A, the main electronic components of RFID tag 1310 include a radio frequency integrated

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circuit (RFIC) 1312 and an antenna 1316. Antenna 1316 is preferably a copper pattern consisting of two portions, although those of skill in the art will recognize that other materials and patterns may be used. Each portion of antenna 1316 is bonded to a thin semi-flexible substrate 1315 which, preferably, is composed of a type of fiberglass that is non-absorptive of the radio signal. RFIC 1312 typically is attached directly to the antenna using a "flip chip" process that is well known to those of skill in the art of integrated circuit fabrication technology. Although not shown in FIG. 8A, as shown in FIGS. 8B and 8C, RFIC 1312 is protectively supported by an epoxy coating 1314. In addition, as shown in FIGS. 8B and 8C, the top surface of RFID tag 1310 is preferably laminated with a plastic coating 1313 to protect antenna 1316. Such RFIC chips mounted on a substrate and sufficiently small for use in the present invention are currently marketed by several vendors, e.g., Intermec Technologies Corporation, Alien Technology Corporation, SCS Corporation, and Hitachi Ltd. Preferably, the RFID tag selected for use includes the capability to be permanently disabled by receipt of a command from an external communicator, so that, for example, the RFID tag can be disabled at checkout. As stated above, the smallest RFID tags operate at the 2.45 GHz radio frequency, which is accepted by most countries of the world for RFID applications.

In accordance with an aspect of the invention, as shown in FIG. 8A, a lateral perforation 1319 is provided near RFIC 1312 so that if both ends of RFID tag 1310 are pulled in opposite directions with sufficient tension, RFID tag 1310 will tear at lateral perforation 1319, thereby separating a significant part of the antenna 1316 from RFIC 1312. This separation renders RFID tag 1310 permanently inoperable. Perforation 1319 in RFID tag 1310 may be offcenter, as it is shown in FIG. 8A. Alternatively, in configurations where RFID chip 1312 is mounted in a hole (not shown) in the substrate 1315 of RFID tag 1310, perforation 1319 is unnecessary because the hole provided for RFIC

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1312 will be the weakest point mechanically of RFID tag 1310 when no perforation 1319 is present. RFID tag 1310 will thus naturally fracture at the location of RFIC 1312 in this configuration. This fracture location, at RFIC 1312, is presently preferred because it results in more complete disablement of RFID tag 1310 since there will no connection whatsoever to at least one of the two portions of antenna 1316. In addition, with this configuration, it is also possible that when the RFID tag 1310 is damaged by opening the bottle, RFIC 1312 could be damaged also contributing to the permanent disablement of the RFID tag 1310.

In a presently preferred embodiment, RFID tag 1310 further includes a protrusion in the form of a cylindrical catch 1318 that is bonded close to an end thereof. Preferably, cylindrical catch 1318 is bonded to RFID tag 1310 on the side opposite to the side on which antenna 1316 is mounted. Cylindrical catch 1318 is preferably formed from a flexible material in a hollow tubular form. As discussed in more detail below, cylindrical catch 1318 is part of the mechanism by which RFID tag 1310 is mechanically disabled when bottle cap 1300 is unscrewed from bottle 1100 for the first time after being sealed at the bottling facility. In an alternative embodiment, RFID tag 1310 includes a slot in substrate 1315 instead of catch 1318. The formation of the slot in this alternative embodiment is discussed in further detail below.

Referring now to FIGS. 9 and 10, bottle insert 1200 includes a central column 1210 having an outside diameter at the surface 1211 that is large enough to cause a tight fit when it is pressed into bottle 1100 (FIG. 7). It is necessary that the fit be sufficiently tight so as to prevent bottle insert 1200 from rotating inside bottle 1100 when cap 1300 is removed for the first time. Optionally, one or more narrow longitudinal cuts 1250 may be made through a wall of central column 1260 to provide some stress relief as bottle insert 1200 is pressed into bottle 1100. Also, there may be two or more radial rib members (not shown) supporting the wall of central column 1260 that allow it to flex

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locally as bottle insert 1200 enters bottle 1100, and then to expand as bottle insert 1200 passes the narrowest diameter of mouth 1110 of bottle 1100. Since most bottles for alcoholic beverages are tapered on the inside, with the orifice being the smallest inside diameter, as seen in FIG. 10A, a shallow narrowing in the area 1270 of central column 1260 may preferably be provided to conform to the tapered orifice of bottle 1100, thus providing increased friction between bottle insert 1200 and bottle 1100. As recognized by those of skill in the art, various other techniques may be used to ensure that bottle insert 1200 fits tightly in mouth 1110 of bottle 1100.

Bottle insert 1200 preferably includes two discs 1220 and 1230 and ratchet wheel 1240 that are pressed or bonded onto the top end of the central column 1260. Discs 1220 and 1230 fit on either side of ratchet wheel 1240. Plain disc 1220 is mounted below ratchet wheel 1240 and acts as a spacer mechanism to separate ratchet wheel 1240 from mouth 1110 of bottle 1100 and to provide a mechanical buffer that prevents damage to ratchet wheel 1240 when bottle insert 1200 is pressed into bottle 1100. Upper plain disc 1230 provides a pouring surface which may preferably be contoured to provide various improvements in pouring the contents from bottle 1100, although these contours are not shown in FIGS. 9 and 10.

As one of skill in the art will readily recognize, although bottle insert 1200 is shown as formed from four parts, bottle insert 1200 may also be manufactured as a single molded part or the central column 1210 may be combined with lower plain disc 1220 and ratchet wheel 1240 to form a single molded component to which top disc 1230 is attached during assembly. Alternatively, as one of skill in the art will readily recognize, one or both of plain discs 1220 and 1230 may also be eliminated. In addition, it is possible that the ratchet wheel or other protrusions could be formed directly on bottle 1100 between threads 1120 and mouth 1110.

Ratchet wheel 1240 operates as a conventional ratchet wheel and

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provides a ratchet action in combination with the catch 1318. Ratchet wheel 1240 allows cap 1300 to be twisted onto bottle 1100, which conventionally has a right-handed thread 1120, because catch 1318 attached to the RFID tag 1310 forces the serrations on ratchet wheel 1240 inward. However, when an attempt is made to remove cap 1300 from bottle 1100, a counterclockwise torque will be applied to cap 1300, pressing catch 1318 against one of the serrations on ratchet wheel 1240. As the torque is increased on cap 1300, catch 1318 will, in effect, pull on one end of RFID tag 1310 while the opposite end of RFID tag 1310 is permanently affixed to cap 1300, eventually causing RFID tag 1310 to break at perforation 1319 (or, in the event that no perforation is provided, at the weakest point of substrate 1316). As one of ordinary skill will readily recognize, the form and number of the ratchets on ratchet wheel 1240 or other protrusions are not significant so long as they do not interact with substrate 1315 when cap 1300 is being installed on bottle 1100 and interact with substrate 1315 when cap 1300 is being removed from bottle 1100. In the alternative embodiment where RFID tag 1310 includes a slot instead of catch 1318, the ratchets on ratchet wheel 1240 or other protrusions extend outward beyond the outer circumference of bottle 1100 at mouth 1110 such that the ratchets or other protrusions contact cap 1300. In this alternative embodiment, the indentation on RFID tag 1310 is formed so that the ratchets on ratchet wheel 1240 or other protrusions pass over when cap 1300 is being installed on bottle 1100, but they do not pass over the indentation when cap 1300 is being removed from bottle 1100. In both embodiments, the result is the same, removal of cap 1300 for the first time generates a lateral force along RFID tag 1310 that results in the fracture of substrate 1318 and total disablement of RFID tag 1310.

FIG. 11 shows how RFID tag 1310 is mounted in cap 1300. Cap 1300 preferably includes a recess 1340 between an end of internal thread 1320 and the inside end 1330. Recess 1340 is only slightly wider than RFID tag 1310.

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During manufacture of cap 1300, RFID tag 1310 is installed in recess 1340 and permanently affixed to cap 1300 at an end 1350 of RFID tag 1310 opposite end at which catch 1318 is mounted, RFID tag 1310 is oriented upon installation such that as cap 1300 is screwed onto bottle 1100, the serrations on bottle insert 1200 pass over catch 1318 and also push RFID tag 1310 into a slight compression against the walls of cap 1300. When cap 1300 is screwed tightly onto the bottle, the serrations on ratchet wheel 1240 will line up longitudinally with the catch 1318 on RFID tag 1310. At some later time, when cap 1300 is unscrewed from bottle 1100 for the first time, the serrations on bottle insert 1200 will snag on catch 1318 as discussed above and the torque on cap 1300 will apply a longitudinal force along the length of RFID tag 1310, essentially stretching RFID tag 1310 along its longitudinal axis and causing RFID tag 1310 to break into two parts, either at perforation 1319, if present, or at some other weak point of RFID tag 1310, e.g., at the RFIC 1312 mounting point. Breaking of RFID tag 1310 into two parts will permanently disable RFID tag 1310 because it will no longer be able to communicate due to a significant change in the characteristics of antenna 1316.

The plastic cap 1300 as previously described is composed of two pieces (1300a,1300b) which are pressed together during assembly. This is because of the need for a recess region 1301 inside the cap. In order for the cap to be released from the injection mold, the cap should be made in two pieces. Those pieces are shown in FIG. 12. The recessed region 1301 does not extend uninterrupted around the inside of the cap. There is a barrier 1302 that prevents the broken part of the tag 1310 from circulating all the way around the recession when the cap is removed from the bottle. This barrier 1302 is visible as hidden lines in FIG. 12b.

The RFID tag 1310 and the recess barrier 1302 are more clearly visible in FIG. 13. The RFID tag may be bonded into the recessed region 1301 of the cap near the barrier 1302 as shown, or it may be locked into place by a narrow

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slot at the time that the two-piece cap is pressed together (not shown).

The tag 1310 has a mechanical coiling bias built into it so that when the tag is fully intact, it attempts to coil radially inward and to spring the catch mechanism inward so that it will be snagged by the ratchet wheel 1240 as the bottle 1100 is opened for the first time.

The barrier 1302 has a taper on the side opposite the secured tag such that if the broken end of the tag 1310 were to travel all the way around the inside of the cap in the recession, the catch mechanism of the tag is forced in an outward radial motion and will be released by the ratchet tooth of the bottle insert.

A "tag collar" is located adjacent radially to the RFID tag so that the broken end of the tag does not fall out of the cap when the cap is removed from the bottle. A small aperture in the collar allows the catch mechanism to protrude into the ratchet mechanism of the bottle insert, but it does not allow the broken segment of tag to slip out.

The catch mechanism 1318 may also have a taper, shown in FIG. 13, and the upper and lower plain discs of the bottle insert assembly may have bevels as shown in FIG. 14. As the cap 1300 is screwed onto the bottle 1100 or off from the bottle, the taper on the catch mechanism and the bevels on the plain discs push the catch mechanism 1318 outwards radially so that the cap can move past the catch mechanism without being hung up.

Although the different aspects of the present invention have been shown and described with respect to preferred embodiments, various changes and modifications can be made which lie within the spirit and scope of the invention. For example, those skilled in the art will recognize that the RFID tag in the second aspect of the invention could be placed in different locations and many other forms of RFID structures could be used. In addition, various types of structures can be used to provide an interconnection between the cap and container such that when the cap is first removed, the RFID tag will become

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disabled. In the first aspect, the tag may be electronically disabled. Thus, numerous changes and modifications can be made while staying within the scope of the invention which is set forth in the appended claims.

CLAIMS

1. An apparatus for use in detecting tampering with a container sealed with a cap, comprising:

a radio frequency transceiver circuit adapted to transmit a signal upon receipt of a transmit command;

an antenna having a fixed length connected to said radio frequency transceiver circuit; and

means for preventing said radio frequency transceiver circuit from transmitting a signal when the container has been tampered with.

2. An apparatus for use in detecting tampering with a container sealed with a cap, comprising:

a substrate having a first portion thereof adapted to be affixed to a portion of the container, and a second portion thereof adapted to be affixed to the cap;

a radio frequency transceiver circuit mounted on said substrate adapted to transmit a signal upon receipt of a transmit command;

an antenna having a fixed length connected to said radio frequency transceiver circuit and mounted on said substrate, adapted to be arranged so that a portion of said antenna is mounted over the container and a portion of said antenna is mounted over the cap such that removal of the cap from the container will cause said antenna to separate thereby preventing said radio frequency transceiver from receiving any commands.

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3. The apparatus of Claim 2, further comprising a security seal mounted over said substrate, said radio frequency transceiver circuit and said antenna.

- 4. The apparatus of Claim 2 or Claim 3, wherein said substrate is positioned such that said antenna forms a magnetic configuration.
- 5. An apparatus for detecting tampering with a container filled with a substance, comprising:

a cap adapted to seal said container when connected thereto;

a radio frequency transceiver circuit adapted to transmit an identification code upon receipt of a command to transmit and mounted within said cap;

an antenna having a fixed length connected to said radio frequency transceiver circuit and mounted to said cap;

a sensor that produces an output indicative of an amount of said substance within said container and mounted within said cap; and

means for disabling said radio frequency transceiver when said sensor indicates a change in said amount of said substance within said container mounted within said cap.

6. The apparatus of Claim 5 wherein said sensor comprises a resistor having a predetermined resistance and a probe connected as a voltage divider and excited by a battery having a fixed voltage, said probe comprising a first conductive rod and a second conductive second rod mounted substantially parallel to each other and perpendicular to a horizontal axis of said cap such that said probe has a low resistance when said first and second rods are in said substance of said container and a high resistance when said probe is not immersed in said substance of said container.

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- 7. An apparatus for detecting tampering with a container filled with a substance, comprising:
 - a cap adapted to seal said container when connected thereto;

a radio frequency transceiver circuit adapted to transmit one of a plurality of identification codes upon receipt of a command to transmit and mounted within said cap;

an antenna having a fixed length connected to said radio frequency transceiver circuit and mounted to said cap;

a sensor that produces an output indicative of an amount of said substance within said container and mounted within said cap; and

means for instructing said radio frequency transceiver to transmit a particular one of said plurality of identification codes based upon said output of said sensor.

8. The apparatus of Claim 7 wherein said sensor comprises a resistor having a predetermined resistance and a probe connected as a voltage divider and excited by a battery having a fixed voltage, said probe comprising a first conductive rod and a second conductive second rod mounted substantially parallel to each other and perpendicular to a horizontal axis of said cap such that said probe has a low resistance when said first and second rods are in said substance of said container and a high resistance when said probe is not immersed in said substance of said container.

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9. A method for use in identifying tampering with a container sealed with a cap, comprising the steps of:

attaching a radio frequency transceiver circuit and an antenna connected thereto to the container, said radio frequency circuit adapted to respond to receipt of a transmit command by transmitting a signal;

disabling said radio frequency circuit when tampering with the container has been detected;

transmitting a transmit command to said radio frequency transceiver circuit; and

identifying tampering with a container by the failure of the container to respond to said transmit command.

- 10. The method of Claim 9, wherein said antenna has a first end connected to the container and a second end connected to the cap, and said step of disabling is accomplished by separating portions of said antenna thereby preventing said radio frequency transceiver circuit from receiving any commands.
- 10 11. An apparatus for use in tracking whether a tax has been paid for a closed container, comprising;
 - a radio frequency identification tag comprising a substrate and a radio frequency transceiver circuit adapted to transmit a signal upon receipt of a transmit command and mounted upon said substrate; and
- a container, said radio frequency identification tag mounted on said container, said radio frequency identification tag containing information indicating that the tax for said container has been paid.
- 12. The apparatus of Claim 11, wherein said information comprises a unique predetermined code.
 - 13. The apparatus of Claim 11 or Claim 12, further comprising:
 a cap for said container having a closed end and an open end, said radio
 frequency identification tag mounted on an inner surface of said cap; and
- an engagement member interconnecting said substrate and said container such that removing said cap from said container results in the fracture of said substrate thereby causing said radio frequency identification tag to become permanently disabled.

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14. The apparatus of Claim 11 or Claim 12, wherein said radio frequency identification tag further comprises an antenna having a fixed length mounted upon said substrate and connected to said radio frequency transceiver circuit and a first interconnection means on said substrate, said apparatus further comprising:

a cap for said container having a cylindrical portion, a closed end of said cylindrical portion and an open end of said cylindrical portion, said radio frequency identification tag mounted on an inner surface of said cylindrical portion of said cap adjacent to said closed end such that said first connection means faces centrally inward; and

- a second interconnection means connected to said container for engaging said first interconnection means when said cap is installed on said container such that removing said cap from said container will exert force on said substrate resulting in the fracture of said substrate thereby causing said radio frequency identification tag to become permanently disabled.
- 15. The apparatus of Claim 14, wherein said first interconnection means is a protrusion affixed to said substrate or a slot in said substrate, and said second interconnection means comprises ratcheting means.

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- 16. A radio frequency identification tag for use in tracking whether a tax has been paid for a closed container comprising:
- a substrate and a radio frequency transceiver circuit adapted to transmit a signal upon receipt of a transmit command and mounted on said substrate, said radio frequency identification tag containing information indicating that the tax for the container has been paid.
- 17. An RFID tag system which communicates with a base station at a predetermined frequency for a container having a metal closure comprising:

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an insulator adapted to be mounted to an exterior surface of said metal closure; and

a radio transceiver system coupled to said insulator comprising

an antenna tuned to said predetermined frequency adapted to be 5 mounted to an exterior surface of the metal closure; and

an RFID IC chip coupled to said antenna and adapted to be coupled to said metal closure.

18. An RFID tag system for communication with a base station at a predetermined frequency comprising:

a metal bottle cap having an interior surface, an exterior surface, and an electrical feedthrough connection from said interior surface to said exterior surface;

an insulator mounted to an exterior surface of said metal bottle cap; a patch antenna mounted to said insulator and coupled to said electrical feedthrough connection at an exterior point, said patch antenna tuned to said predetermined frequency; and

an RFID IC chip mounted on an interior surface of said metal bottle cap and coupled to said patch antenna via said feedthrough connection and to said metal bottle cap.

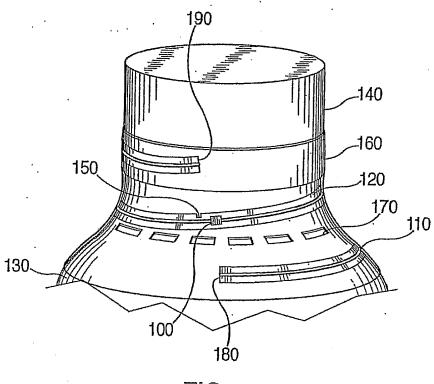
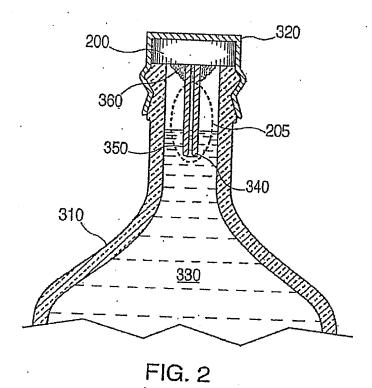
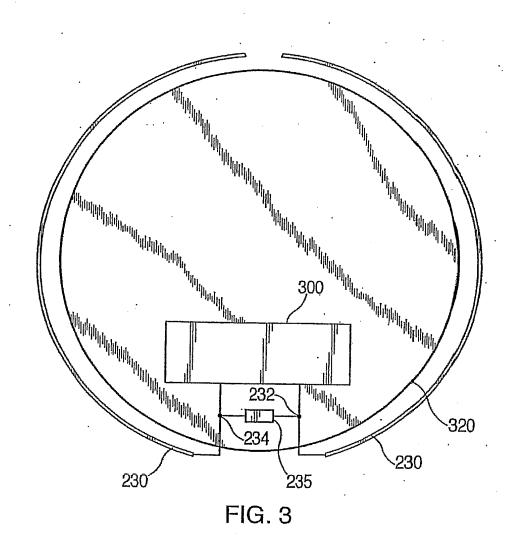
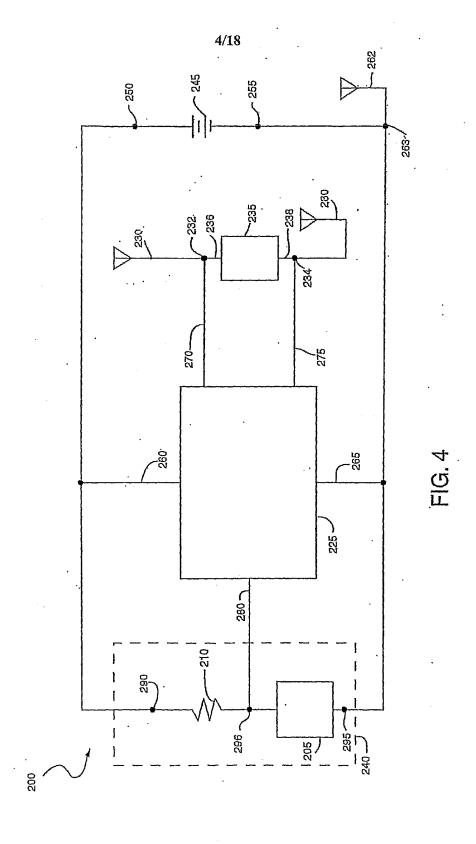


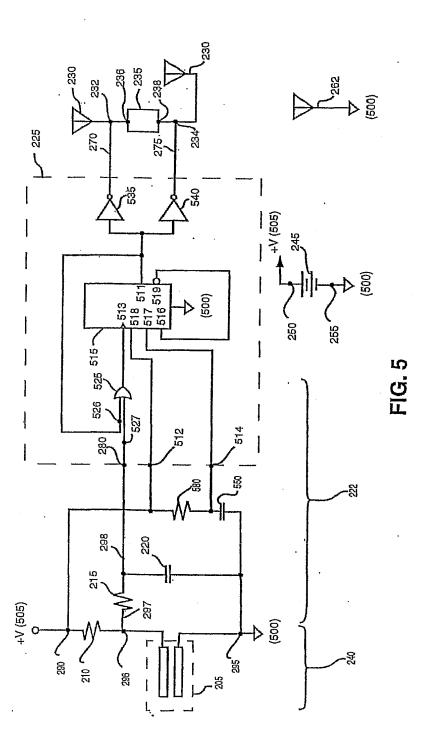
FIG. 1

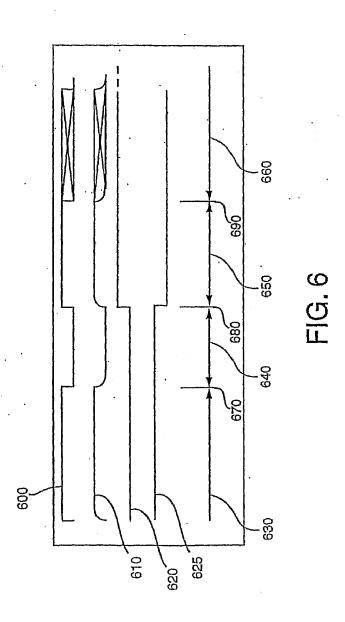
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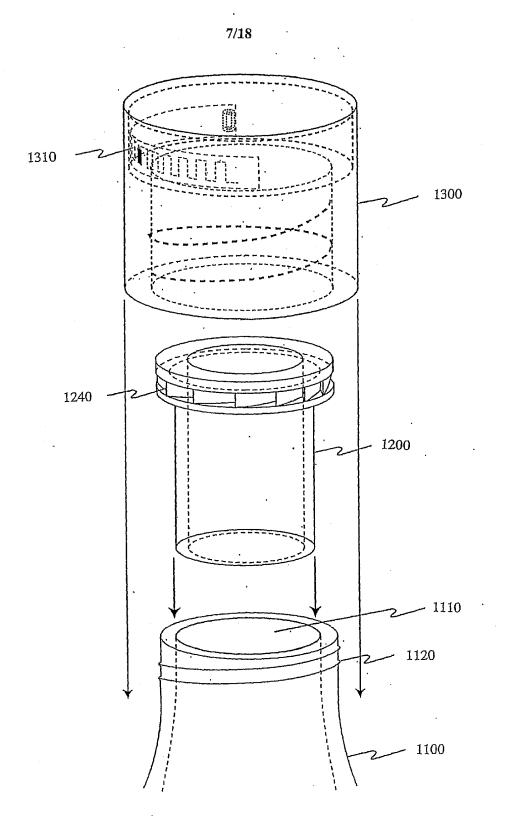


FIG. 7A

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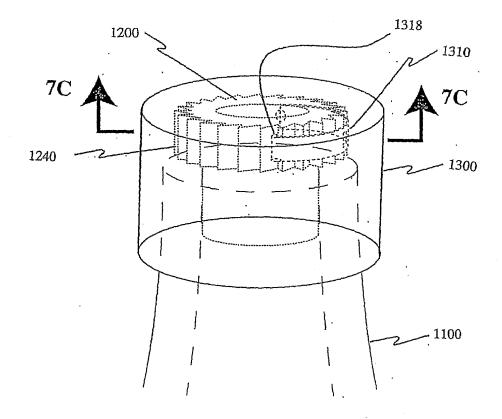


FIG. 7B

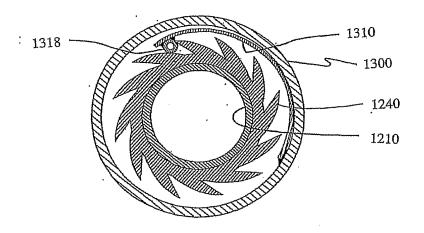


FIG. 7C

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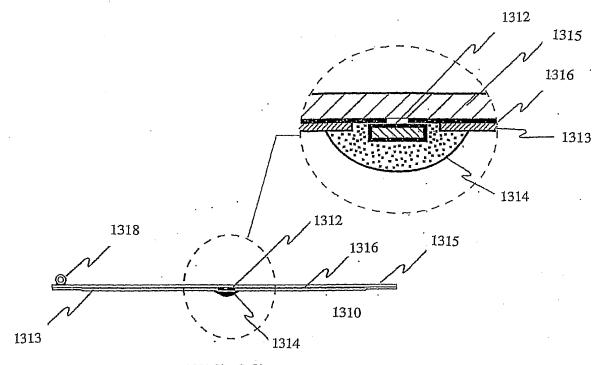


FIG. 8C

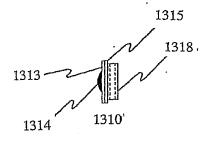
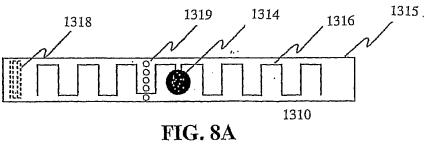
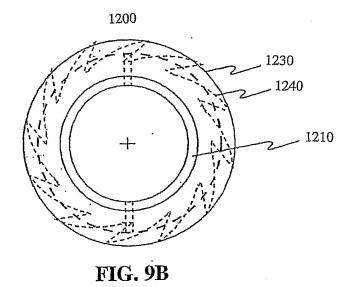


FIG. 8B





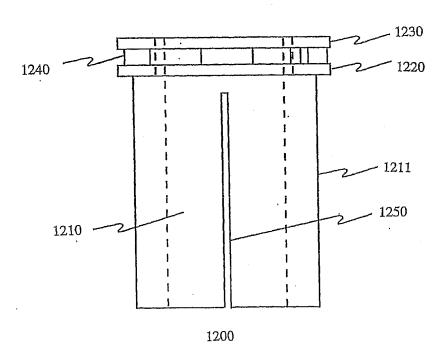


FIG. 9A

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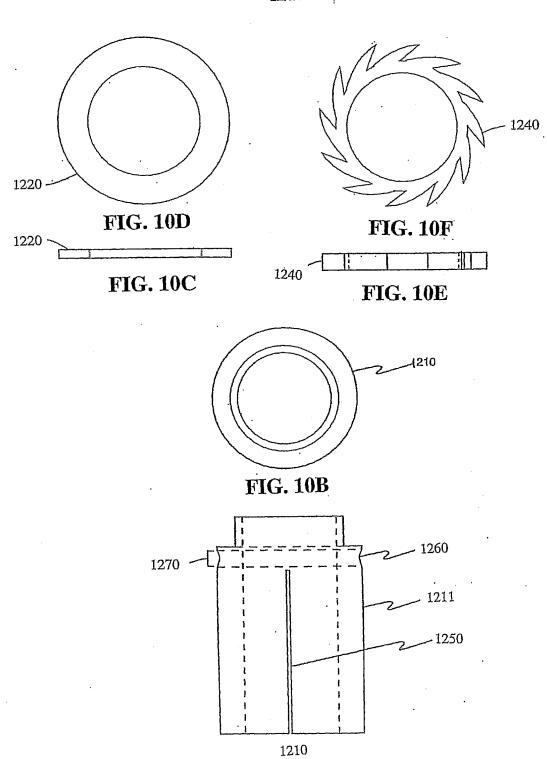


FIG. 10A

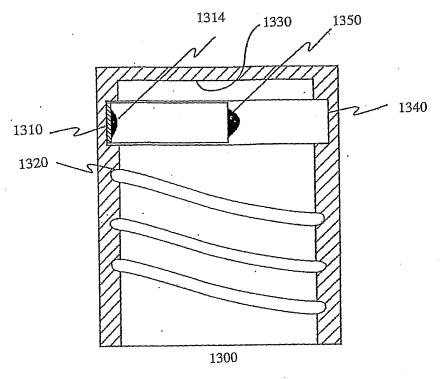


FIG. 11B

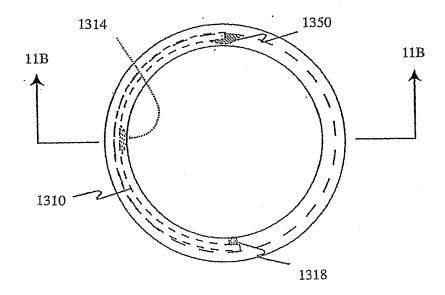


FIG. 11A

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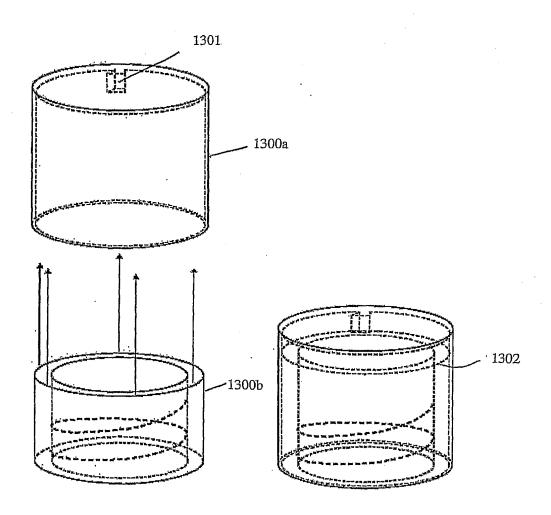
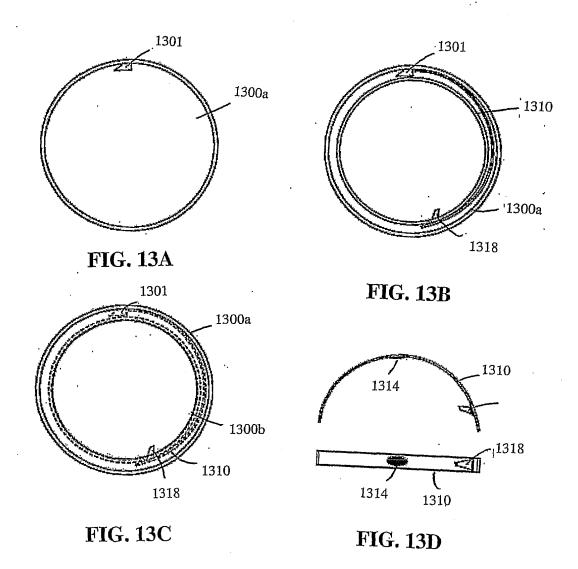


FIG. 12A

FIG. 12B



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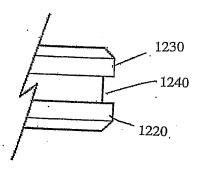


FIG. 14

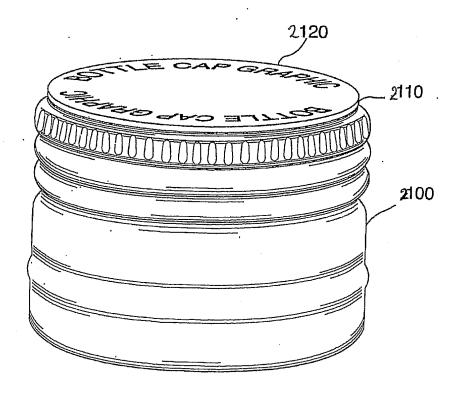


FIG. 15

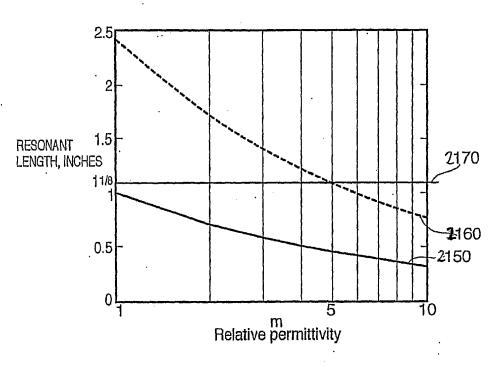
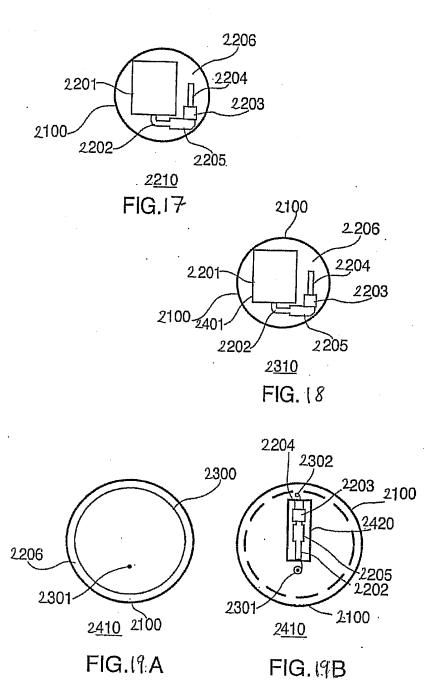


FIG. 16

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2 567 106

21) N° d'enregistrement national :

85 10261

(51) Int CI4: B 65 D 47/20; A 47 K 5/13.

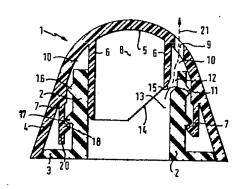
② DEMANDE DE BREVET D'INVENTION

A1

- 22) Date de dépôt : 4 juillet 1985.
- 30 Priorité : DE, 6 juillet 1984, nº P 34 24 940.0.
- 43 Date de la mise à disposition du public de la demande : BOPI « Brevets » n° 2 du 10 janvier 1986.
- 60 Références à d'autres documents nationaux apparentés :

- (71) Demandeur(s): LAUBE Werner. DE.
- (72) Inventeur(s): Werner Laube,
- 73 Titulaire(s):
- (74) Mandataire(s) : Cabinet Beau de Loménie.

- 64 Couvercle tournant d'un contenant.
- d'un contenant 3. L'orifice de prélèvement 9 qui en fait partie débouche sur une gorge circulaire 10 formée entre deux épaulements cylindriques creux 12, 13 concentriques, profilés sur la paroi interne 5 du couvercle 1. Afin d'obtenir un canal de passage 11 précis et réglable entre l'intérieur du contenant 3 et l'orifice de prélèvement 9, il est prévu un premier tronçon de canal 12 dans la paroi du goulot 2 et un deuxième tronçon de canal 13 dans la paroi de l'épaulement cylindrique creux central 6. Une rotation du couvercle 1 par rapport au goulot 2, et donc une orientation des tronçons de canal 12, 13 l'un par rapport à l'autre, permet de fermer et d'ouvrir, entièrement ou partiellement, le canal de passage 11.



"Couvercle tournant d'un contenant"

La présente invention concerne un couvercle tournant destiné au goulot d'un contenant, en particulier un contenant à savon liquide orientable à suspendre, comportant un orifice de prélèvement débouchant sur une gorge circulaire formée entre deux épaulements cylindriques creux concentriques profilés sur la paroi interne du couvercle, ainsi qu'un goulot s'emboîtant de manière étanche dans la gorge circulaire, jusqu'à une profondeur prédéfinie.

Un couvercle fileté de ce type est décrit dans DE-OS 26 47 750. 10 Le couvercle fileté connu possède un orifice de prélèvement qui débouche sur une gorge circulaire, laquelle reçoit l'extrémité du goulot du contenant, située sur la face interne du couvercle. La gorge circulaire est limitée intérieurement par un épaulement en forme d'anneau à insérer dans le goulot du contenant et extérieurement par un deuxième épaulement cylindrique creux 15 de la paroi interne du couvercle. Le deuxième épaulement cylindrique creux peut être glissé de manière étanche, par le biais de sa face interne, sur au moins une nervure circulaire entourant le goulot du contenant, près de l' orifice d'écoulement, et est pourvu d'un filet femelle près de son bord libre;à ce dernier, correspond un filet mâle au goulot du contenant. Le 20 mouvement axial réalisé par rotation du couvercle dans le filet, par rapport au goulot du contenant et à son orifice, est limité par des butées axiales. Etant donné que l'épaulement annulaire central de la face interne du couvercle est d'une hauteur essentiellement plus faible que celle de l' épaulement cylindrique creux périphérique, un mouvement axial correspondant 25 du couvercle par rapport au goulot du contenant permet d'obtenir un orifice de passage entre les extrémités libres de l'épaulement cylindrique creux central et le bord du goulot du contenant, vers la gorge circulaire et par conséquent vers l'orifice de prélèvement.

Dans le contenant du type connu, l'orifice de sortie est formé
30 par une discontinuité du bord du goulot du contenant, laquelle est
réalisée dans toute l'épaisseur de la paroi. Cet orifice ne peut être
confectionné à l'aide d'outils de calibrage du type classique. De plus,
un dosage de la quantité de substance n'est pas possible lors de son
écoulement, étant donné qu'après dévissage du couvercle, l'épaulement
35 annulaire central, lequel est inséré initialement de manière étanche dans
le goulot, libère toute la périphérie du goulot du contenant.

L'objet de la présente invention est de développer, pour un contenant déterminé destiné à être couché, en particulier pour une bouteille, une fermeture fonctionnelle intégrée en une pièce , laquelle n'est pas 40 nécessairement du type à visser, à rabattre ou à basculer, qui permet le prélèvement du produit que le contenant renferme et qui peut être fabriquée

à un prix coûtant relativement bas. Il est en particulier conçu un couvercle dont l'orifice de prélèvement peut être ouvert en partie ou entièrement par simple rotation, de préférence par le biais d'un dispositif d'accouplement exempt de filet, selon un angle d'orientation déterminé. A cet effet, il y a lieu , de manière préférentielle, d'attribuer un angle d'orientation déterminé du couvercle, sans équivoque pour l'utilisateur, définissant la limite entre la position d'ouverture et la position de fermeture du couvercle par rapport à l'orifice de prélèvement.

En ce qui concerne le couvercle tournant du type mentionné au début,

à monter sur le goulot d'un contenant, la solution selon l'invention se
caractérise en ce qu'il est obtenu, lors de la rotation du couvercle par rapport
au goulot du contenant, un canal de passage entre l'orifice de prélèvement et
l'intérieur du contenant, par le biais de la réalisation d'un alignement entre
un premier tronçon de canal formé dans la paroi du goulot du contenant et

un deuxième tronçon de canal formé dans la paroi de l'épaulement cylindrique
creux central. Suivant l'invention, le bord du goulot est par conséquent
aminci uniquement intérieurement par le premier tronçon de canal et soutenu
extérieurement par la paroi interne du couvercle.

Les longueurs individuelles des tronçons du canal, mesurées dans le 20 sens longitudinal du goulot, seront de préférence respectivement plus petites et la somme des longueurs individuelles de ces tronçons de canal sera plus grande que la longueur totale du canal de passage.

Lorsque les tronçons de canal de la fermeture tournante suivant l'
invention, comportant le couvercle et le goulot du contenant, subissent une
25 rotation l'un par rapport à l'autre de manière à ce qu'il n'existe aucun
alignement entre eux, même partiel, le passage entre l'intérieur du contenant
et l'orifice de prélèvement est fermé. Par contre, lorsqu'il est réalisé un
alignement partiel ou complet des tronçons de canal, le produit du contenant
peut être, via le canal de passage ainsi formé entre l'épaulement cylindrique
30 creux central et le goulot du contenant, amené vers l'extérieur à travers l'
l'orifice de prélèvement du couvercle.

Le couvercle tournant suivant l'invention est de préférence utilisé en tant que dispositif de fermeture destiné à un "distributeur" de savon liquide. A cet effet, le contenant est habituellement articulé autour d'un axe horizontal de manière à ce que l'orifice du contenant soit amené vers le bas. L'épaisseur ou la grosseur de la projection s'échappant du dispositif, lors des basculements alternés, se règle au moyen du système de fermeture selon l'invention, en amenant les deux tronçons de canal en alignement partiel ou complet, ou en positionnant, par le biais d'une rotation du couvercle par rapport au goulot, l'un ou l'autre tronçon de canal en travers, sur la valeur souhaitée.

Selon une autre caractéristique de l'invention, le premier tronçon de canal peut consister en une rainure pratiquée sur la face interne du goulot. La rainure peut être réalisée sur l'épaisseur totale du goulot; toutefois, afin de ne pas exercer un effet déstabilisateur et de permettre l'apport d'un joint périphérique sur la face externe du goulot, la rainure sera de préférence prévue sur une partie uniquement de l'épaisseur de la paroi du goulot. En outre, la largeur du premier tronçon de canal, mesurée dans le sens circonférentiel du goulot, sera au moins égale à la largeur de l'orifice de prélèvement, mesurée dans le sens circonférentiel. Lorsque l'angle d' ouverture de l'orifice de prélèvement, mesuré par rapport à l'axe du cylindre creux, est d'environ 60°, il peut être avantageux d'étendre la largeur du premier tronçon de canal à un angle d'ouverture d'environ 90°.

Le deuxième tronçon de canal, lequel relie le cas échéant l' orifice de prélèvement à l'intérieur du contenant, est formé de préférence 15 en raccourcissant l'épaulement cylindrique creux central dans une zone périphérique comprenant l'orifice de prélèvement. Dans une forme de construction préférentielle, l'épaulement cylindrique creux central est raccourci par voie d'une coupe en biais par rapport à l'axe du cylindre, en particulier sur à peu près une moitié de l'extrémité libre de l'épaulement, 20 de manière à obtenir un passage continu sur toute la longueur. Par exemple, il peut être pratiqué une coupe d'une inclinaison adaptée, permettant ainsi de réaliser une jonction avec le premier tronçon de canal, en partant approximativement du centre de l'extrémité libre circulaire (à l'origine) de l'épaulement cylindrique creux central, de manière à ce qu'un "quartier" 25 inférieur de l'épaulement cylindrique soit supprimé. Une telle coupe en biais par rapport à l'axe du cylindre permet, par rotation du couvercle par rapport au goulot du contenant, d'augmenter progressivement de zéro ou d'un minimum vers un maximum et de diminuer de nouveau progressivement vers zéro le mouvement d'ouverture et de fermeture du canal de passage, lorsque le 30 couvercle est sujet à une rotation selon un angle correspondant à l'angle d'ouverture du premier tronçon de canal.

A la jonction entre le premier tronçon de canal et le deuxième tronçon de canal, la hauteur maximale du canal de passage, mesurée dans le sens axial du cylindre, sera environ égale à la profondeur du premier tronçon de canal réalisé à la tête du goulot et de préférence environ égale à la profondeur de la rainure pratiquée sur la face interne du goulot. En position ouverte du couvercle, un écoulement régulier du produit à travers les canaux alignés peut alors avoir lieu, sans qu'il se produise de refoulement remarquable.

40 En vue d'une manipulation aisée du couvercle selon l'invention, il peut enfin être avantageux de prévoir une butée de limitation de la plage de

rotation du couvercle par rapport au goulot, et donc de la position d'ouverture et de fermeture du couvercle, sur le goulot du contenant et sur la paroi du couvercle entourant le goulot. Il peut par exemple être avantageux de concevoir les butées de limitation de la position d'ouverture et de fermeture entre un ergot sur la face externe du goulot et un évidement pratiqué dans le sens circonférentiel du cylindre à l'extrémité libre de l'épaulement cylindrique périphérique. Au lieu d'un seul ergot sur la face externe du goulot, il peut également y être prévu deux ergots; en même temps, un seul contre-ergot solidaire du couvercle suffit éventuellement. Des arrêts de ce type permettent de définir et de limiter l'angle de rotation du couvercle par rapport au goulot. Le cas échéant, il en résulte une manipulation simplifiée du dispositif de fermeture et en particulier une plus grande facilité de recherche de réglages déterminés.

A ce propos, il y a lieu de préciser que l'angle d'ouverture, mesuré par rapport à l'axe des épaulements cylindriques creux, de l'orifice de 15 prélèvement, du premier tronçon de canal, ainsi que de l'évidement prévu à l'extrémité libre de l'épaulement cylindrique creux périphérique, s'élèvera progressivement, de préférence à environ 60°, environ 90° et environ 180° minimum. Cette distribution de valeurs et une largeur radiale de l'orifice de prélèvement d'un ordre de grandeur de l,0 mm permettent d'obtenir, lors d'une utilisation du système en tant que dispositif de fermeture d'un "distributeur" de savon liquide, des résultats de fonctionnement particulièrement avantageux.

Afin de réunir le couvercle et le goulot, il est prévu, de manière préférentielle, dans le système de fermeture selon l'invention, un assemblage 25 à emboîtement. L'assemblage doit évidemment permettre une rotation du couvercle par rapport au goulot. Le goulot du contenant possède de préférence un rebord d'arrêt circulaire, tandis que l'épaulement cylindrique creux périphérique est pourvu, sur sa face interne, d'une butée d'emboîtement circulaire correspondante, destinée au rebord d'arrêt. En même temps, il peut 30 être avantageux de protéger la zone située entre le goulot du contenant et les parois internes de la gorge circulaire formée entre l'épaulement cylindrique creux central et l'épaulement cylindrique creux périphérique, contre l' infiltration ou la pénétration de produit. La face externe de l'épaulement cylindrique central et la face interne du goulot seront de préférence ajustées 35 l'une par rapport à l'autre. Il peut toutefois être prévu, sur la face externe du goulot du contenant, des nervures circulaires dont la crête s' ajuste de manière étanche à la paroi interne de l'épaulement cylindrique creux périphérique. Afin de faciliter l'emboîtement mentionné ci-dessus et de renforcer l'étanchéité de fonctionnement de l'ensemble, le goulot du contenant 40 et/ou les parties contiguës du couvercle peuvent par exemple être fabriqués dans une matière élastique souple et compressible.

Oes particularités de l'invention sont exposées par la suite à l'aide de la représentation schématique en annexe : Il y est représenté :

Figure 1 - une coupe, verticale à l'axe du cylindre, du dispositif de fermeture en position ouverte ;

Figure 2 - une vue du dessus du couvercle ;

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Figure 3 - une coupe à travers un couvercle en position fermée, emboîté sur un goulot de contenant; ladite coupe étant effectuée après une rotation de 90° de la figure l ;

Figure 4 - une vue du dessous des éléments vus en coupe suivant la figure 3 ;

Figure 5 - une coupe à travers le goulot du contenant ;

Figure 6 - une vue de côté du goulot du contenant; et

Figure 7 - une vue du dessus du goulot du contenant suivant la figure 5 ou 6.

Le dispositif de fermeture suivant l'invention, représenté au moyen des figures 1 à 7, consiste en un couvercle désigné dans son ensemble par le repère 1 et en un goulot, désigné dans son ensemble par le repère 2, d'un contenant 3. Le couvercle 1 est constitué de la coiffe 4, au sens étroit, comportant un épaulement cylindrique creux central 6 et un épaulement cylindrique creux périphérique 7 profilés sur la face interne. Les épaulements cylindriques creux ainsi que la coiffe 4, de préférence, sont concentriques et possèdent un même axe de cylindre 8. Un orifice de prélèvement 9 est prévu dans la coiffe 4. Cet orifice de prélèvement 9 débouche dans la gorge circulaire 10 formée entre les deux épaulements cylindriques creux 6 et 7 de la face interne 5 de la coiffe 4. L'orifice de prélèvement 9 peut avoir une largeur de 1,0 mm par exemple, mesurée dans le sens radial, et peut s'étendre sur 60° par exemple dans le sens circonférentiel (Figure 2).

Suivant l'invention, la jonction entre l'orifice de prélèvement 9 et l'intérieur du contenant 3 est réalisée par le biais d'un canal de

30 passage désigné dans son ensemble par le repère 11, lequel consiste en un premier tronçon de canal 12 et un deuxième tronçon de canal 13. Le premier tronçon de canal 12 se situe dans la paroi du goulot 2, le deuxième tronçon de canal 13 est formé dans la paroi de l'épaulement cylindrique creux central 6, et plus précisément à la place d'une partie de ladite paroi. Le canal de

35 passage 11 entre l'orifice de prélèvement 9 et l'intérieur du contenant 3 peut être ouvert ou fermé par le biais d'un déplacement l'un par rapport à l'autre des tronçons de canal prévus respectivement dans le couvercle 1 et le goulot 2. Afin de réaliser le deuxième tronçon de canal 13, l'épaulement cylindrique creux central 6 peut être raccourci dans une zone périphérique 40 renfermant l'orifice de prélèvement 9, de manière à ce que, lors d'un

déplacement du couvercle 1 par rapport au goulot 2, les tronçons de canal 12 et 13 soient au moins alignés et forment un canal de passage 11.

Oans l'exemple de réalisation, l'épaulement cylindrique creux central 6 est raccourci par le biais d'une coupe 14 en biais par rapport à l'axe du cylindre 8, en particulier sur à peu près une moitié de l'extrémité libre de l'épaulement 6, de manière à obtenir un passage ininterrompu, mais néanmoins infléchi sur toute la longueur. En définitive, il ne s'agit plus d'un canal (13) au sens étroit, mais d'un large orifice de jonction 15 avec le premier tronçon de canal 12. Toutefois, dans l'esprit de l'invention, un tel orifice est considéré comme canal ou tronçon de canal. La hauteur du canal de passage 11 à la jonction 15 entre le premier et le deuxième tronçon de canal 12,13, mesurée dans le sens axial du cylindre 8, sera au maximum environ égale à la profondeur de la rainure pratiquée sur la face interne du goulot 2; ladite rainure représentant, dans l'exemple de réalisation, le premier tronçon de

Le goulot 2 du contenant 3, destiné à être introduit dans la gorge circulaire 10 entre l'épaulement cylindrique creux central 6 et l'épaulement cylindrique creux périphérique 7, peut posséder sur sa face extérieure une lèvre d'étanchéité circulaire 16 et un rebord d'arrêt circulaire 17. Au rebord d'arrêt 17 doit correspondre une butée 18 également circulaire sur la face interne de l'épaulement cylindrique creux périphérique 7, de manière à ce que le couvercle l puisse être emboîté à l'aide de la butée 18 sur le rebord d'arrêt 17. De plus, le goulot 2 peut posséder sur sa face extérieure un ergot d'arrêt 19 (Figure 6), lequel offre la possibilité, conjointement avec une 25 extension 20 saillant à l'extrémité inférieure de l'épaulement cylindrique creux périphérique 7, de limiter la rotation du couvercle l par rapport au goulot 2 à un angle d'orientation déterminé et d'assurer un arrêt en position fermée du système, lors de sa rotation mécanique.

Dans la position de rotation relative, suivant la figure 1, en

30 basculant le contenant 3 en position tête en bas, le produit qu'il contient
peut s'écouler vers l'extérieur, dans le sens de la flèche 21, par le passage
formé par les tronçons de canal 13, 12 et l'orifice de prélèvement 9. Lors d'
une légère rotation du couvercle 1, à partir de la position ouverte, en
particulier dans la plage de 90° de la largeur circonférentielle du premier

35 tronçon de canal 12, la hauteur du passage 15, mesurée dans le sens axial du
cylindre 8, entre le deuxième tronçon de canal 13 et le premier tronçon de
canal 12 est diminuée et, par conséquent, lors d'un bref basculement du
contenant, l'écoulement du produit dans le sens de la flèche 21 est atténué
proportionnellement. Lors d'une rotation plus importante du couvercle 1 autour

40 de l'axe 8, et dès lors un déplacement plus important de l'épaulement
cylindrique creux central 6 par rapport au premier tronçon de canal 12, de

sorte que le passage 15 se trouve entièrement obturé, le produit ne peut plus s'écouler du contenant 3. Oans l'exemple de réalisation,ce cas a lieu au plus tard lorsque le couvercle 1 a subi une rotation d'environ 90° par rapport au goulot 2 du contenant 3.

Liste des repères de référence

- l = Couvercle
- 2 = Goulot
- 3 = Contenant
- 4 = Coiffe
- 5 = Paroi interne (4)
- 6 = Epaulement central
- 7 = Epaulement périphérique
- 8 = Axe du cylindre
- 9 = Orifice de prélèvement
- 10 = Gorge circulaire
- ll = Canal de passage
- 12 = Premier tronçon de canal
- 13 = Oeuxième tronçon de canal
- 14 = Coupe
- 15 = Jonction ou.passage (12/13)
- 16 = Lèvre d'étanchéité
- 17 = Rebord d'arrêt
- 18 = Butée
- 19 = Ergot d'arrêt
- 20 = Extension
- 21 = Sens de la flèche

REVENOICATIONS

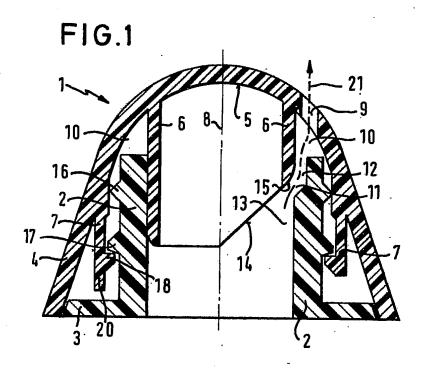
- Couvercle tournant (1) destiné au goulot (2) d'un contenant (3), en particulier un contenant à savon liquide orientable à suspendre, comportant un orifice de prélèvement (9) débouchant sur une gorge circulaire (10) formée
 entre deux épaulements cylindriques creux concentriques (6, 7) profilés sur la paroi interne (5) du couvercle, ainsi qu'un goulot (2) s'emboîtant de manière étanche dans la gorge circulaire (10), jusqu'à une profondeur prédéfinie, caractérisé en ce qu'il est obtenu, lors de la rotation du couvercle (1) par rapport au goulot (2), un canal de passage (11) entre 1' orifice de prélèvement (9) et l'intérieur du contenant (3), par le biais de la réalisation d'un alignement entre un premier tronçon de canal (12), formé dans la paroi du goulot (2), et un deuxième tronçon de canal (13) formé dans la paroi de l'épaulement cylindrique creux central (6).
- 2. Couvercle suivant la revendication l, caractérisé en ce que les longueurs individuelles des tronçons de canal (12, 13), mesurées dans le sens de l'axe longitudinal du goulot (8), sont respectivement plus petites et la somme des longueurs individuelles plus grande que la longueur totale du canal de
- Couvercle suivant la revendication l ou 2, caractérisé en ce que le
 premier tronçon de canal (12) consiste en une rainure pratiquée sur la face interne du goulot (2).
- Couvercle suivant une ou plusieurs des revendications 1 à 3, caractérisé en ce que la largeur du premier tronçon de canal (12), mesurée dans le sens circonférentiel du goulot (2), est au moins égale à la largeur de l'orifice
 de prélèvement (9), mesurée dans le sens circonférentiel.
 - 5. Couvercle suivant une ou plusieurs des revendications 1 à 4, caractérisé en ce que le deuxième tronçon de canal (13) est formé en raccourcissant l'épaulement cylindrique creux central (6) dans une zone périphérique comprenant l'orifice de prélèvement (9).
- 30 6. Couvercle suivant la revendication 5, caractérisé en ce que l'épaulement cylindrique creux central (6) est raccourci par voie d'une coupe (14) en biais par rapport à l'axe du cylindre (8), en particulier sur à peu près la moitié de l'extrémité libre de l'épaulement (6), de manière à obtenir un passage continu et ininterrompu sur toute la longueur.
- 7. Couvercle suivant les revendications 3 et 6, caractérisé en ce que la hauteur maximale du canal de passage (11), mesurée dans le sens axial du cylindre (8), à la jonction (15) entre le premier tronçon de canal (12) et le deuxième tronçon de canal (13) est environ égale à la profondeur de la rainure pratiquée sur la face interne du goulot (2), ladite rainure formant
- 40 le premier tronçon de canal (12)

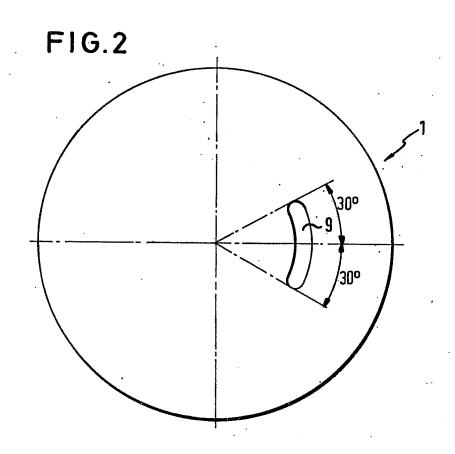
passage (11).

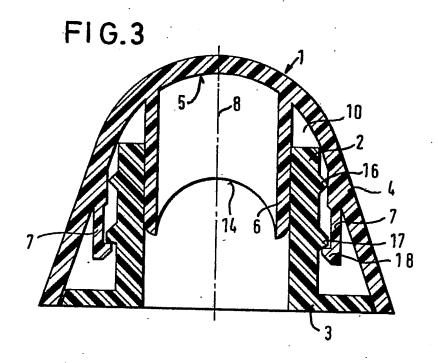
- 8. Couvercle suivant une ou plusieurs des revendications 1 à 7, caractérisé en ce qu'il est prévu une butée de limitation (19, 20) de la rotation du couvercle (1) par rapport au goulot (2), sur le goulot et sur la paroi du couvercle entourant le goulot, en particulier entre un ergot d'arrêt (19)
- sur la face externe du goulot (2) et une extension (20) à l'extrémité libre de l'épaulement cylindrique creux périphérique (7).
 - 9. Couvercle suivant une ou plusieurs des revendications 1 à 8, caractérisé en ce que l'angle d'ouverture, mesuré par rapport à l'axe du cylindre (8), de l'orifice de prélèvement (9), du premier tronçon de canal (12) et la
- largeur circonférentielle de l'extension (20) servant d'arrêt, prévue à l'extrémité libre de l'épaulement cylindrique creux périphérique (7), s'élèvent progressivement à environ 60°, environ 90° et environ 180° minimum.

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- 10. Couvercle suivant une ou plusieurs des revendications l à 9, caractérisé en ce qu'il comporte un assemblage à emboîtement entre le couvercle (1) et le goulot (2).
- 11. Couvercle suivant la revendication 10, caractérisé en ce que le goulot (2) du contenant (3) possède un rebord d'arrêt (17) circulaire et en ce que l'épaulement cylindrique creux périphérique (7) présente une butée d'emboîtement (18) circulaire sur sa face interne, destinée au rebord d'arrêt (17).
- 12. Couvercle suivant une ou plusieurs des revendications l à ll, caractérisé en ce que le goulot (2) du contenant (3) possède au moins une lèvre d'étanchéité (16) circulaire sur sa face externe; ladite lèvre d'étanchéité s'adaptant parfaitement à la face interne de l'épaulement cylindrique creux périphérique (7).







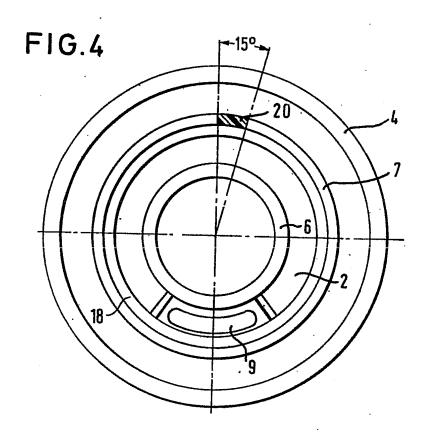


FIG.5

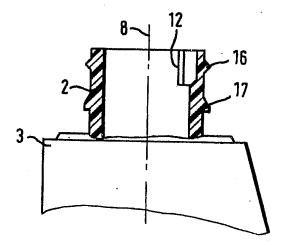
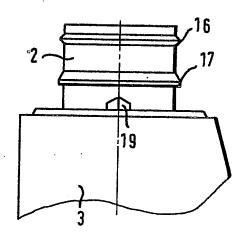


FIG.6



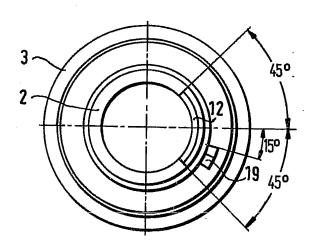


FIG.7

PATENT SPECIFICATION

(11) 1298 084

084

NO DRAWINGS

(21) Application No. 62919/69 (22) Filed 24 Dec. 1969

(31) Convention Application No. 182 544 (32) Filed 31 Dec. 1968 in

(33) France (FR)

(45) Complete Specification published 29 Nov. 1972

(51) International Classification A61K 9/00, 9/04

(52) Index at acceptance A5B 763 764 B8C A



(54) CAPSULES AND OTHER SELF-SUPPORTING MEDICINAL PACKAGING MEANS

(71) We, SOCIETE D'ETUDES, DE RECHERCHES ET D'APPLICATIONS SCIENTIFIQUES ET MEDICALES (E.R.A.S.M.E.) a French Company of 67, Avenue De Wagram, 75-Paris 17E, France and RENE CLAUDE, a citizen of France, of 13, Rue Des Hauts-Closeaux, 92-Sevres (Hauts De Seine), France, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to capsules and like self-supporting medicinal dosage forms comprising a shell formed of thermoplastic and to processes for producing the same.

Gelatine capsules have been in use for many years as a medicinal dosage form. It has recently been suggested that gelatine be replaced by certain other polymeric substances such as hydroxyalkylcelluloses. In both these cases the production process consists of immersing a capsule-forming mould into a solution of the polymeric substance (gelatine or hydroxyalkylcellulose), withdrawing the mould from the solution so as to obtain, on the mould surface, a substantially uniform coating of the solution, and drying this coating to form a self-supporting film or skin which constitutes a capsule.

It is also known that coated tablets or powders may be produced by impregnating tablets or powders with a solution or dispersion of at least one polymeric substance, and then drying the impregnated tablets or powders to form a film of the polymeric substance(s) covering the tablets or powders.

As will have been appreciated the processes described in the two preceding paragraphs are "wet" processes. They incur substantial disadvantages which may be eliminated or reduced by the present invention.

It has been suggested to produce threads or packagings for medical use by plastic conversion of polyvinyl alcohol, but it was found that plastic conversion of polyvinyl alcohol by conventional processes for conversion of plastics materials produced a product which was of low solubility or insoluble, and which was brittle and not suitable for use in capsules.

The present invention renders possible the industrial production of self-supporting medicinal dosage forms such as capsules which may be administered together with medicines which they contain and which dissolve, disintegrate or become porous within the human body, thereby releasing the medicines.

The invention provides a process for producing capsules and like pharmaceutical dosage forms having a medicinally-inert shell comprising shaping by fusion in the absence of a solvent at least one thermoplastics selected from the following specified materials to form a medicinally-inert shell having sufficient thickness so that it is self supporting.

(a) Vinylpyrrolidone/vinyl acetate copolymers

(b) Polyacrylic acid;(c) polymethacrylates;

(d) polyoxyethylene having a molecular weight between 600,000 and 4,000,000;

(e) hydroxypropyl cellulose; and

(f) polyvinyl alcohol having an ester index higher than 200.

These thermoplastics should be pharmaceutically acceptable. In some cases, they should dissolve or disintegrate in a selected region of the digestive tract. They should above all be workable and able to be formed to shape whilst in the plastic state by industrial processes which allow the production of capsules and packaging means in very great numbers.

The shaping may be performed by injection, moulding, heat-forming or depositing and fusing powder on an arbour.

It is envisaged that the capsules or other self-supporting dosage forms may be administered orally, rectally or vaginally and the shell will dissolve, disintegrate or become porous under the action of the physiological

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[Price 25p]

environment thereby releasing medicament contained in the shell.

A thermoplastics usable according to the invention should be non-toxic, and should (in some cases) dissolve or disintegrate in a selected region of the digestive tract. They must also complementarily be able to withstand conversions and forming to shape in the molten or fused state so that the self-supporting capsules or other packaging means can be produced on an industrial scale. This is the reason why, among the thermoplastics corresponding to the above criteria, the applicants have chosen a particular group of thermoplastics according to the invention, these thermoplastics may be employed singly, or mixed with each other, or with additives, such as plasticisers, antioxidants and release agents.

According to the invention, it is thus possible to employ vinylpyrrolidone/acetate copolymers. The preferred copolymer has a vinylpyrrolidone/vinyl acetate ratio of 60:40, a density of approximately 1.27 and a melting point of approximately 165°C. These copolymers are water soluble within a very wide pH range; they are preferably employed with plasticisers, among which may be mentioned dioctyl phthalate, glycerin and sorbitol and with release agents such as stearamide, oleamide and calcium stearate, and with conventional anti-oxidants. They may be used alone or mixed with other plastics materials applicable according to the invention, in particular with polyvinyl alcohols having a higher ester index than 200, and polyacrylic acid, or even with other compatable plastics materials such as cellulose polyacetate. The copolymers of vinylpyrrolidone and of vinylacetate may be applied according to the invention by means of the different processes specified, in which the fusion of the product plays a part; the tests performed have shown however that these coplymers were more particularly appropriate for the injection-moulding process.

It is equally possible to employ polyacrylic acid alone or mixed with additive such as plasticisers, anti-oxidants and/or agents. Polyacrylic acid may be mixed with other thermoplastic materials according to the invention and more specifically with the copolymers of vinylpyrrolidone and vinyl acetate. This polyacrylic acid usable for the production of self-supporting capsules or other packaging means according to the invention and whose melting point is of approximately 120 to 135°C, has the property of being soluble in a weakly alkaline environment and insoluble in an acid environment; it is thus possible by application of this polymer, to produce gastro-resistive and entero-soluble capsules or packaging means. Packaging means possessing properties of this nature are unknown at present.

Polymethyacrylates are equally applicable according to the invention. The preferred

methacrylate polymers are: polybutylmethacrylate and polydimethylaminoethyl methacrylate which is soluble in an acid environment and is consequently suitable for the production of desage forms which are soluble er dispersible in the stomach, and the copolymers of methyl methacrylate and of methacrylic acid which, depending on their methacrylic acid content, become soluble in a basic environment and consequently become viable for the production of capsules or packaging means which do not dissolve or disintegrate except in an intestinal environment. As with the other polymeric substances applicable according to the invention these polymethacrylates and copolymers may be employed mixed with different additives and with different other polymeric substances such as polyoxyethylenes or polyvinyl acetate.

It is equally possible to employ the polyoxyethylenes having a molecular weight between 600,000 and 4,000,000. Such polymers proved to be particularly appropriate for application by the heat-moulding process even without employing plasticisers. To increase the rigidity of the material, fillers having a reinforcing action such as silica, kaolin or chalk may be incorporated into the polyoxyethylenes. The polyoxyethylenes may be mixed with other plastic materials such as polyacrylic or polycarboxyvinyl substances, with which it forms complex polymeric substances.

A hydroxypropylcellulose having a mean molecular weight Mw = 275,000 is equally employed. This material simultaneously has the properties of solubility thermoplasticity and surface-activity. Owing to this fact, it is wholly suitable for injection moulding owing to its qualities of fluidity when hot, and of antiadhesion capacity. It has been observed that 105 the grades having a high molecular weight (Mw=900,000) are brittle, and those having a low molecular weight (Mw=75,000) are soft. Thanks to the self-lubricating nature of the substance, plasticising is unnecessary. The 110 incorporation of an anti-oxidant, for example Irganox 1076 (0.2%) prevents discolouration of the product at high temperature.

It has been discovered that hydroxypropylcellulose is equally appropriate for extrusion 115 of plates and for heat-moulding. It is compatible, in particular, with polyoxyethylene, the other cellulose derivatives, and gelatine.

Finally, according to the invention, it is equally possible to employ a polyvinyl alcohol 07I having a high ester index, of the order of 200 to 300, or a molecular degree of hydrolysis of 70 to 80%. The polymer in the form of powder of a fineness between 100 and 200 microns, is deposited by means of a spraygun 125 of a fluidised bed on an arbour heated to 320°C. The grade of powder most suitable for this application has a density of 1.27 to 1.30 and a melting point of approximately 130°C. To prevent thermal degradation of the 130

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polyvinyl alcohol employed in this process, it is appropriate to protect the same by incorporation of up to 0.4% of "Irganox 1076". It has been observed that the release agents which are surface active as a rule, should be excluded from the composition of the mixtures, owing to the fact that they raise the risk of delaying the agglomeration of the plastic powders and their fusion. To improve the fluidity of the polymeric substances in the molten state, a plasticiser (5%) such as glycerine or propyleneglycol, may be added to the polymer.

If it is intended, for production of selfsupporting capsules and dosage forms, to employ polyvinyl alcohol, it is necessary to make an appropriate choice of the initial polymeric substance and equally of the process for conversion of this initial material. If these choices are inappropriate and do not correspond to the invention, packaging elements which are insoluble or non-disintegrable are frequently obtained, since the initial material has undergone a definite number of chemical conversion such as, for example reticulations.

25 The invention is illustrated by the following Examples.

Example 1

In a mixer of the "belt" type, heated to 60°C, a mixture of the following ingredients was prepared:

Polyvinylpyrrolidone/vinyl acetate copolymer (density 1.27, melting point 165°C) 100 g Stearamide (release agent). 6 parts by weight Irganox 1076 (anti-oxidant) 0.4 Dioctyl phthalate (plasticiser) 30

Capsules were produced by injection moulding of this mixture with a piston press heated to 170°C, using a pressure of 1000 kgs/cm². The capsules were rigid; smooth and shiny, and had a length of 16.7 mms, a diameter of 6.6 mms and a wall-thickness of 0.1 mm.

Sheets having a thickness of 1 mm were produced on an extrusion press equipped with a flat (orifice) die-plate, which were employed to produce capsules by heat-moulding.

Example 2

A homogenising operation was performed in a bladed mixer on a mixture having the following composition: Polyacrylic resin (density 1.2 melting point 122°C—135°C) 100 g Stearamide (release agent) 4 parts by weight Irganox 1076 (anti-oxidant) 0.4

Capsules were made by injection moulding from this homogenised mixture on a piston press, at 180°C and at a pressure of 1200 kgs/cm². The capsules were accurately dimensioned.

Sheets of the homogenised mixture having

a thickness of 1 mm, a width of 180 mms, were extruded by means of the extrusion press used in Example 1. The sheets were smooth, uniform and incurred little transversal contraction. The heating temperatures were: 150°C in the barrel, 170° at the head, 180°C at the die-plate. Heat-moulding was applied on these sheets to produce capsules. Tubes having a diameter of 6.65 mms and a wallthickness of 1.1 mm were extruded on the same extrusion press equipped with a tubedrawing die on this occasion. These were cut into sections of 16.70 mms and moulded at one extremity by means of a heating electrode having the shape of a spherical cap, to produce capsules.

Example 3

A mixer of the "belt" type, heated to 50°, was employed to produce a mixture of two polymers in equal proportions (50/50): polyacrylic resin and polyvinylpyrrolidone/vinyl acetate copolymer.

Polyacrylic resin Polyvinylpyrrolidone/vinyl	50	g	0=
acetate copolymer Stearamide (release agent) IRGANOX 1076 (anti-oxidant) Dioctyl phthalate (plasticiser)	50 3 0.2 20	ත හ හ හ	85

A moulding operation was performed after homogenisation under heat, at a temperature of 170°C. Plates were obtained, which were smooth, uniform, homogeneous, rigid but not brittle.

An extrusion press equipped with a flat nozzle was employed to extrude sheets from this mixture, which had a thickness of 1 mm, were homogeneous, non-adhesive, and of smooth appearance.

The conditions of extrusion were the following:

Heating temperatures — barrel: 165°C — Head: 180,C — Nozzle: 170°C.

Self-supporting capsules and dosage forms were produced by heat-moulding from the sheets obtained by compression or extrusion.

Example 4

A paddle mixer was used to prepare a mixture consisting of: Polymethacrylate of butyl and of 110 dimethylaminoethyl (melting point 122°C) 100 g Stearamide (release agent) 4 parts by weight Irganox 1076 (anti-oxidant) 0.4 ", ",

Injection mouldings were produced from this mixture on a piston press at 160°C and at a pressure of 1200 kgs/cm2. Capsules having the aforesaid dimensions were obtained. An extrusion press equipped with a flat nozzle as specified above, was employed with the same mixture to extrude sheets having a thickness of 0.5 mm and of 1 mm, and a width of

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160 mms. These sheets were shiny and transparent. Capsules were produced by heatmoulding from these sheets.

Tubes having the aforesaid dimensions were extruded on the same extrusion press, equipped with a tube-drawing die on this occasion. After cooling and calibrating in a calibrating tool at the temperature of 56°C, the tubes were cut into sections of 16.70 mms and 10 moulded at one end by means of a heating electrode having the shape of a spherical cap, to obtain self-supporting capsules.

The conditions of tube extrusion were: Heating temperature of the extrusion press: Barrel: 130°C — Head 150°C — Die:

140°C. Speed of extrusion: 5 m/minute.

Plates having a thickness of 1 mm, which were shiny and transparent, were equally moulded from the mixture by compression at a temperature of 160°C and under a pressure of 300 kgs/cm2. Self-supporting capsules were produced by heat-moulding from the plates.

Example 5

A paddle mixer was used to prepare a mix-25 ture consisting of:

Polyoxyethylene (melting point 70° -100 g molecular weight 4,000,000) Stearamide (anti-adhesive

5 parts by weight agent) 30 Irganox 1076 (anti-oxidant) 0.15 ,, ,, ,, Titanium oxide (filler)

Capsules having the aforesaid dimensions were injection-moulded from this mixture at a temperature of 200° under a pressure of 1300 kgs/cm². The capsules retained their initial shape after storage. As an alternative, an extrusion press equipped with a flat nozzle whose characteristics have been mentioned above, was equally employed to extrude sheets of the mixture, having a thickness of 1 mm and of 1.5 mm, from which were produced capsules by heat-moulding by means of a plunger of a length of 16.70 mms.

Example 6

A paddle mixer was used to prepare a mixture consisting of:

Hydroxypropylcellulose (molecular weight 275,000; softening point:

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100 g 130°C) 4 g Stearamide (release agent) Butyl hydroxyanisol (anti-oxidant) $0.02 \, \mathrm{g}$

Capsules were injection-moulded at a pressure of 880 kgs/cm² and at a temperature of 180°C. The capsules obtained were rigid, transparent, shiny and incurred little contraction and no deformation at all and were not affected by humidity after being stored for 48 hrs. in an ambient atmosphere. The capsules were not easily breakable.

Example 7

A "band" mixer was employed to prepare: Polyvinyl alcohol (ester index 270, density 1.2, melting point 130°C) 4 parts by weight Stearamide (release agent) Irganox 1076 (2) 11 (anti-oxidant) 22 22 Dibutyl phthalate 10 (plasticiser) Titanium oxide (filler) 70 23

Capsules were injection moulded from this mixture on a piston press at 190°C, and under a pressure of 1300 kgs/cm2. The capsules had a length of 16.70 mms, a diameter of 6.65 mms, and a wall-thickness of 1/10 mm. As an alternative, this same mixture was extruded in sheet form by means of an extrusion press having a screw of a diameter of 25 mms and a length equal to 25 diameters, equipped with a flat nezzle having a die-slot of 200 mms length and a width of 2 mms. The heating temperatures of the extrusion press were: 140°C in the barrel, 175°C at the head, and 170°C at the nozzle. Sheets were obtained, having a thickness of 1mm, and a width of 150 mm. The sheets were then formed into capsules. Plates having a thickness of 1 mm were moulded by compression from the same, nonplasticised, mixture at the temperature of 170°C and under a pressure of 270 kgs/cm². The plates were rigid, transparent, slightly brittle but not adhesive and possessed the following mechanical characteristics:

Breaking strain: 350 kgs/cm² — elongation: 180%.

The plates thus obtained were converted by stamping to a depth of 16.70 mms and capsule shells formed. The same mixture was deposited by fluidisation on a punch previously heated to 280°C. The powder became agglomerated on the punch to form a self-supporting shell.

Example 8

A film of polyvinyl alcohol obtained from a substance having a mean ester index exceeding 200 was heated to approximately 140°C, was heat-moulded to produce dosage forms having a diameter of approximately 10 mms and a depth of approximately 5 mms. The forms disintegrated easily in water and were 110 suitable for packaging medicines which should be released from their packages in the stomach.

Different capsules or dosage forms were also made with the following mixtures:

polyvinyl alcohol polyvinyl acetate		% % %	115
polyvinyl alcohol polyvinyl acetate		%	
glycerine	-	% %	120
polyvinyl alcohol butyl methacrylate	7.5	,-	120
polydimethylaminoethylmeth- acrylate	7.5	%	

30

The result obtained with the different capsules or dosage forms thus produced are similar to those obtained from the film of polyvinyl alcohol described above.

5 Example 9

A powder consisting of vinylpyrrolidone/
vinyl acetate copolymer containing 40% of
vinyl acetate was fed into a mould-heated to
approximately 100°C (that is to say to the
softening point of the copolymer). A cold
punch was inserted into the mould and a small
cylindrical capsule was formed by flowage and
and agglomeration of the powder. The capsule had a diameter of 6.5 mms and a depth
of 17 mms and one end of it was open.

The capsule was water-soluble.

Mixtures of the same copolymer with plasticisers or colourants or fillers of various kinds, such as other plastics materials such as carboxyvinyl polymers, copolymers of ethylene and of maleic anhydride, may be employed according to the same technique.

Example 10

The solubilities of capsules produced in the foregoing Examples were measured in different environments. The different environments were: water, artificial gastric juices, and artificial intestinal juices, at a temperature of $37^{\circ}\text{C} \pm 2^{\circ}\text{C}$. The capsules were exposed to continuous vertical agitation. The periods of dissolution of the capsules were measured.

The formulae applied in the production of the secretions were:

NaCl pepsin HCl distilled water to	2 g 3.2 g 7 ml 1000 ml	35
artificial intestinal monopotassium phosphate pancreatine 0.2 N NaOH distilled H ₂ O	. jurices 6.8 g 10 g 190 ml 600 ml	40

		Periods of Dissolution			
	Nature of the Capsules	water	artificial gastric secretions (pH 1)	artificial intestinal secretions (pH 7.5)	Remarks
1)	polyvinyl alcohol	12 minutes	15 minutes	20 minutes	complete dissolution
2)	polyoxyethylene	15 ,,	12 ,,	10 ,,	,,
3)	polyacrylic	insoluble	insoluble	85 "	partial dissolution
4)	polymethacrylate of butyl and dimethyl- aminoethyl	2 hours	5 minutes	2 hours	complete dissolution
5)	polyvinylpyrrol- idone/vonyl acetate	12 minutes	15 "	10 minutes	complete dissolution
6)	PVP-VA/polyacrylic	2 hours	low solu- bility	45 mins.	partial dissolution
7)	hydroxypropyl- cellulose	10 mins.	20 mins.	15 mins.	complete dissolution

The solubility test was performed on the apparatus specified in USP XVI standard for tests covering disintegration of tablets.

WHAT WE CLAIM IS: -

1. A process for producing capsules and like 50 pharmaceutical dosage forms having a medicinally-inert shell comprising shaping by fusion in the absence of a solvent at least one thermoplastic selected from the following specified materials to form a medicinally-inert shell having sufficient thickness so that it is self supporting:

- (a) vinylpyrrolidone/vinyl acetate copolymers
- (b) polyacrylic acid;
- (c) polymethacrylates;

0.5

б0

30

5

(d) polyoxyethylene having a molecular weight between 600,000 and 4,000,000;

- (e) hydroxypropyl cellulose; and

(f) polyvinyl alcohol having an ester index higher than 200.

2. A process as claimed in claim 1, wherein the thermoplastic contains at least one additive selected from plasticizers, release agents, antioxidants and fillers.

3. A process as claimed in claim 1 or 2, wherein the thermoplastic is a vinylppyrrolidine/vinyl acetate copolymer and the shaping is performed by injection-moulding.

4. A process as claimed in claim 1 or 2, wherein the thermoplastic is polyacrylic acid and the shaping is performed by heat-moulding

5. A process as claimed in claim 1 or 2, wherein the thermoplastic is a homopolymer

or copolymer of methacrylate and the shaping is performed by hot-forming of an extruded thin-walled tube.

6. A process as claimed in claim 1 or 2, wherein the thermoplastic is polyoxyethylene and the shaping is performed by heat-mould-

ing.

7. A process as claimed in claim 1 or 2, wherein the thermoplastic is hydroxypropyl cellulose and the shaping is performed by injection-moulding.

8. A process for producing self-supporting medicinal packaging means in encapsulated form substantially as described herein with reference to any one of Examples 1 to 9.

9. Self-supporting medicinal packaging means in encapsulated form produced by a process as claimed in any one of claims 1 to 8.

MARKS & CLERK

Printed for Her Majesty's Stationery Office by the Courier Press, Learnington Spa, 1972. Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

UK Patent Application (19) GB (11) 2 136 782 A

(43) Application published 26 Sep 1984

- (21) Application No 8407186
- (22) Date of filing 20 Mar 1984
- (30) Priority data
 - (31) 8307830
- (32) 22 Mar 1983
- (33) GB
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- (51) INT CL³ B65D 41/34
- (52) Domestic classification B8T 120B 13A TC
- (56) Documents cited GB A 2003765 GB 1507539 GB 1473482

GB 1312206 GB 1209395 GB 0651238

(58) Field of search B8T

(54) Pilferproof closure

(57) A pilferproof closure made from malleable metal having a top and a depending skirt has a circumferentially extending slit 13 interrupted by frangible bridges 14 to form a retaining band 15 below the slit. The retaining band has cut on its inner surface a circumferentially extending score line 16 and/or a set of axial score lines 17 extending from the free edge of band towards the slit for making it more difficult to tamper with the closure without leaving visible sign of the tampering.

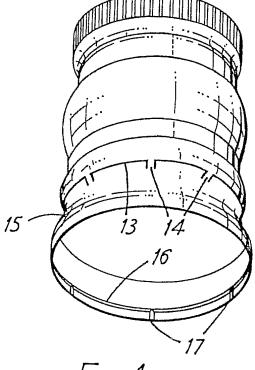
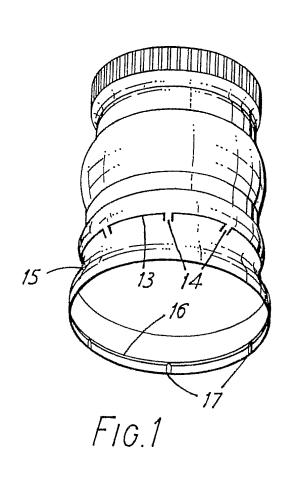
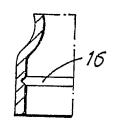
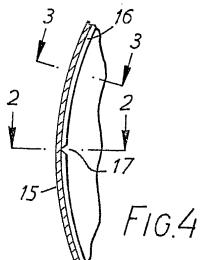


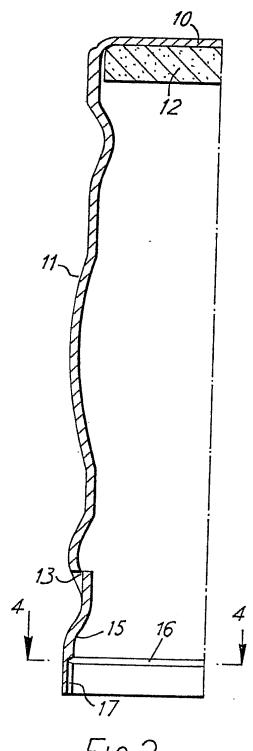
FIG.1



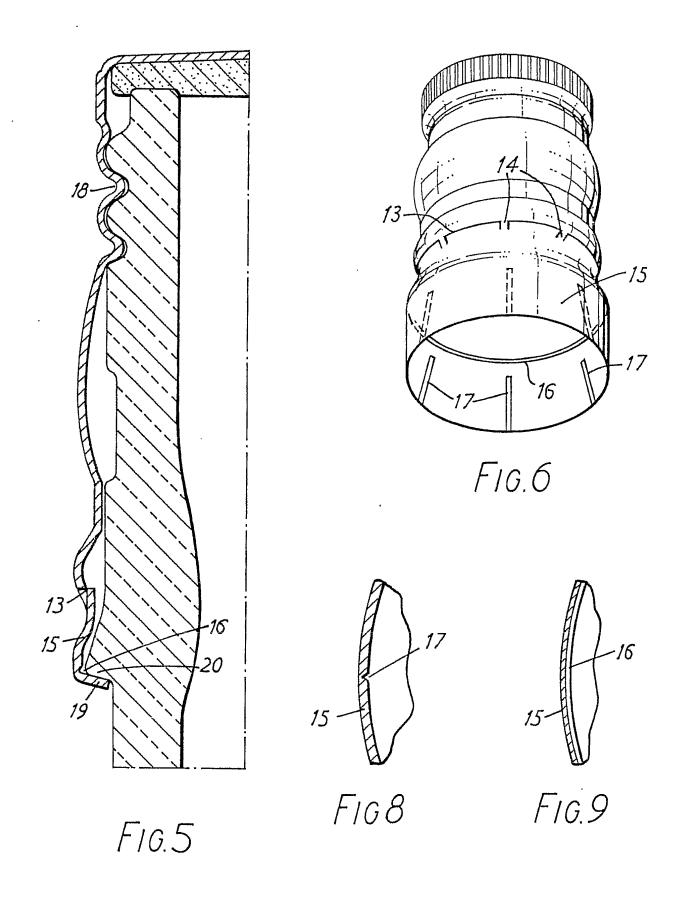


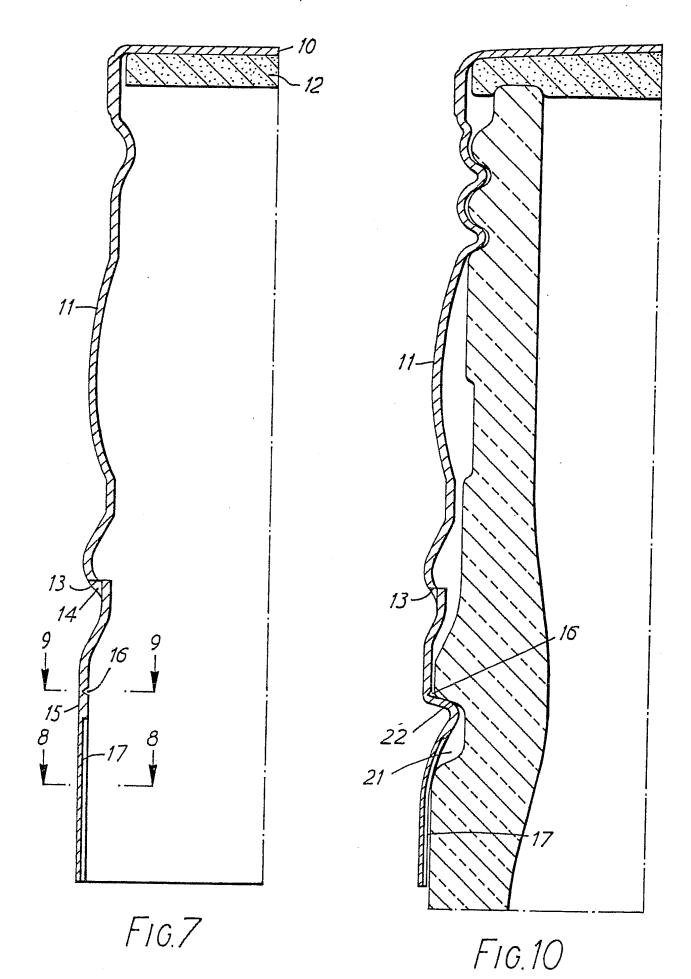
F16.3





F16.2





SPECIFICATION

Improvements relating to pilfer-proof closures

5 This invention relates to closures and more particularly to closures of the pilfer-proof type which are made from a malleable metal such as aluminium or aluminium alloy and which incorporate a slit extending circumferentially of the skirt of the closure, the 10 slit being interrrupted by frangible bridges at angularly spaced intervals about the lengthwise axis of the closure. The part of the skirt below the skit is known as the security band. On application of the closure to the screwlthreaded neck of a container 15 such as a bottle, a part of the skirt above the level of the circumferential slit is rolled into conformity with the screw-thread on the bottle and the security band is substantially fixed against axial movement towards the top of the bottle. Such fixing is commonly 20 achieved by deforming the free edge portion of the band under the bottom edge of a peripheral shoul-

deforming an axially intermediate part of the band into contact with at least the upper edge portion of a 25 peripheral groove moulded in the external surface of the neck. When the closure is unscrewed the bridges are fractured and the security band is left on the bottle, whence it may subsequently be removed. However such closures have been found to have

der moulded on the external suface of the neck or by

30 been tampered with by using a spatula or a similar bladed tool to prise the said parts of the security band away from the surface of the bottle suficiently far to enable the closure to be unscrewed without fracturing the bridges, the closure subsequently

35 being carefully re-applied so that there is no obvious sign that the closure has been tampered with. According to the invention in one aspect there is provided a closure for application to the screwthreaded neck of a container, comprising a top and a

40 peripheral skirt depending from the top, a slit extending circumferentially of the skirt and interrupted at angularly spaced intervals by frangible bridges, and a circumferentially extending score line cut on the radially-inner surface of the skirt at a

45 location axially intermediate the slit and the free edge of the skirt.

According to the invention in another aspect there is provided a closure for application to the screwthreaded neck of a container, comprising a top and a peripheral skirt depending from the top, a slit extending circumferentially of the skirt and interrupted at angualrly spaced intervals by frangible bridges, a plurality of axially-extending score lines cut on the radially inner surface of the skirt and extending from the free edge of the skirt towards said slit and a circumferentially extending score line cut on the radially-inner surface of the skirt at a location axially intermediate the slit and the free edge of the skirt

60 In still another aspect the invention provides a closure for application to the screw-threaded neck of a container, comprising a top and a peripheral skirt depending from the top, a slit circumferentially of the skirt and interrupted at angularly spaced inter-65 vals by frangible bridges, and a plurality of axially-

extending score lines cut on the radially inner surface of the skirt and extending from the free edge of the skirt towards said slit.

Where the axial score lines are provided, it is 70 preferred that there should be at least six such lines equiangularly spaced about the lengthwise axis of the closure.

Where the axial and circumferential score lines are provided in combination, the axial score lines may 75 either intersect or stop short of the circumferential score lines.

Where, as is usual, a closure is designed to fit a particular container neck form, the circumferential score line will prefereably be positioned so as, when applied to the container, to be located at a point of stress concentration where the scurity band is deformed inward under the upper wall portion of a peripheral groove or the lower edge of a peripheral shoulder on the container.

5 Some embodiments of the invention will now be described by way of example with reference to the accompanying drawings in which:

Figure 1 is an underneath perspective view of a first closure according to the invention prior to 90 application to a container.

Figure 2 is a local sectional elevation on the line 2-2 of Figure 4,

Figure 3 is a local sectional elevation on the line 3-3 of Figure 4.

Figure 4 is a fragmentary plan view on the line 4-4 of Figure 2,

Figure 5 is a local sectional view elevation showing the closure of Figures 1 to 5 applied to the neck of a bottle,

100 Figure 6 is a perspective view from underneath of an applied second closure according to the invention.

Figure 7 is a local sectional elevation of the closure of Figure 6,

105 Figures 8 and 9 are respectively fragmentary sectional plan views on the lines 8-8 and 9-9 respectively of Figure 7, and

Figure 10 is a local sectional view showing the closure of Figure 6 applied to the neck of a bottle.

110 Referring to first to Figures 1 to 5, the closure shown is made from a malleable metal alloy and comprises a tap 10 and a depending skirt 11. A wadding gasket 12 is located against the underside of the top. Spaced above the free bottom edge of the skirt is a circumferential slit 13 interrupted by say ten or twelve frangible bridges 14 so that the bottom portion of the skirt forms a security band 15 in the well-known manner. A circumferentially extending score line 16 is cut on the radially-onner surface of

120 the band at a location spead above the bottom edge of the band, and six or more axial score lines 17 are cut to extend along the radially inner surface of the band from the free edge of the band to the score line 16

The score line 16 is so positioned that when the closure is hown applied to the bottle as shown in Figure 5, a screw-thread 18 having been formed on the upper portions of the skirt in the well-known manner the score line 16 is disposed at the location
 of a sharp inward bend formed on the bottle where

30 groove in the neck.

the bottom edge portion 19 of the security band is deformed inwardly under a peripheral shoulder 20 in the neck of the Bottle. The score line 16 is thus disposed at a location of high stress concentration resulting from the said deformation.

Any attempt to prise the portion 19 away from the shoulder is likely to result in tearing along one or more of the score lines 17, but if the attempt is successful the straightening and the necessary sub10 sequent re-forming of the band at the location of the score line 16 results in the occurrence of a fracture at the line 16 which thus reveals the tampering.

Figures 6 to 10 show an application of the invention to another form of pilfer-proof closure 15 which is made from malleable metal alloy and in which the security band is much deeper than in the construction of Figures 1 to 5 and is secured against axial movement towards the mouth of the bottle by being bent inward at the location of the sharp upper 20 edge of a peripheral groove 21 in the external surface of the neck, so that the security band portion is deformed into engagement with at least the upper wall portion 22 of groove 21. Again the security band has a circumferential score line 16 and six or more 25 evenly spaced axial extending score lines 17 cut in its radially inner surface and may extend half way through the thickness of the metal. In this instance, the score lines 17 stop short of the score line 16 but extend from the bottom edge of the band into the

The ideal number of score lines 17 on a 31mm closure is thought to be six, but the number will vary with the size of the closure. Thus it is thought that the score lines should be in the range 10 to 25mm 35 apart and preferably in the range 15 to 20mm apart. The prising of the security band outward away from the neck strains the metal adjoining the side edges of the tool and if a score line is adjacent either or both side edges of the tool the chances of tearing the 40 metal at the location of one or other of the score lines are increased because there is then only a narrow band of unweakened metal between a score line 17 and the side edge of the tool to share in absorbing the strain. However, if the axial score lines 45 are too close together, tampering with the security band may result in the score lines bending open, enabling the closure to be re-applied without leaving evidence of tampering.

Even if the attempt to circumvent the proper 50 operation of the score lines 17 is successful, the person tampering with the closure still has to contend with the score line 16, which operates in the same way as described in Figure 5 to show evidence of the attempted tampering.

In alternative constructions, a circumferential score line 16 alone is provided, positioned as to be at a point of stress concentration as described above.

In other alternative constructions the circumferential score line 16 is omitted and only the axial score 60 lines 17 are provided.

It will be understood that the invention is applicable to all forms of metal closures where a pilfer-proof band is provided, and that the shape and form of the closure above the band is immaterial.

CLAIMS

- A closure for application to the screwthreaded neck of a container, comprising a top and a peripheral skirt depending from the top, a slit extending circumferentially of the skirt and interrupted at angularly spaced intervals by frangible bridges and a circumferentially extending score line cut on the radialy-inner surface of the skirt at a location axially intermediate the slit and the free edge of the skirt.
- A closure for application to the screwthreaded neck of a container, comprising a top and peripheral skirt depending from the top, a slit
 extending circumferentially of the skirt and interrupted at angularly spaced intervals by frangible bridges, and a plurality of axially-extending score lines cut on the radially inner surface of the skirt and extending from the free edge skirt of towards said
 slit.
- A closure for application to the screwthreaded neck of a container, comprising a top and a peripheral skirt depending from the top, a slit extending circumferentially of the skirt and interputed at angularly spaced intervals by frangible bridges, a plurality of axially-extending score lines cut on the radially inner surface of the skirt and extending from the free edge of the skirt towards said slit and a circumferentially extending score line cut on the radially-inner surface of the skirt at a location axially intermediate the slit and the free edge of the skirt.
- 4. A closure as claimed in claim 2 or claim 3, wherein the said axially extending score lines num 100 ber at least six and are equiangularly spaced about the lengthwise axis of the closure.
 - 5. A closure as claimed in claim 3 or claim 4 when appendant to claim 3, wherein the axial score lines stop short of the circumferential score line.
- 105 6. A closure as claimed in any one of claims 2 to 5, wherein the axial score lines are 15mm to 20mm apart.
- In combination, a container having a screwthread on its neck and, below the thread a surface
 facing generally in the opposite direction to the mouth of the container and extending about the neck, and a closure made from malleable metal engaged on said thread, said closure having a top and a peripheral skirt depending from the top, a slit
 extending circumferentially of the skirt at a level below the screw-thread, which slit is interrupted at angularly spaced intervals by frangible bridges, and a circumferentially extending score line cut on the
- 120 between the slit and the free edge of the skirt at or adjacent which location a sharp inward bend is formed in the skirt to cause the skirt to lie against said surface of the container.

radially inner surface of the skirt at a location

- 8. The combination claimed in claim 7 wherein 125 the skirt has formed on its inner surface a plurality of axial score lines extending from the free edge of the skirt towards said circumferentially-extending score line.
- 9. In combination, a container having a screw-130 thread on its neck and, below the thread a surface

facing generally in the opposite direction to the mouth of the container and extending about the neck, and a closure made from malleable metal engaged on said thread, said closure having a top and a peripheral skirt depending from the top, a slit extending circumferentially of the skirt at a level below the screw-thread, which slit is interrupted at angularly spaced intervals by frangible bridges, a portion of the skirt below the slit being formed inward to lie against said surface, and the skirt having on its inner surface a plurality of axial score lines extending from the free edge of the skirt towards said circumferentially-extending score line.

- 10. A closure for application to the screw-15 threaded neck of a container, which closure is substantially as hereinbefore described with reference to and as illustrated in Figures 1 to 4 or in Figures 6 to 9 of the accompanying drawings.
- 11. The combination, with a container having a 20 screw-threaded neck, of a closure substantially as hereinbefore described with reference to and as illustrated in Figure 5 or in Figure 10 of the accompanying drawings.

Printed in the UK for HMSO, D8818935, 7/84, 7102. Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

UK Patent Application (19) GB (11) 2 158 424 A

(43) Application published 13 Nov 1985

- (21) Application No 8506919
- (22) Date of filing 18 Mar 1985
- (30) Priority data (31) 21612
- (32) 20 Apr 1984

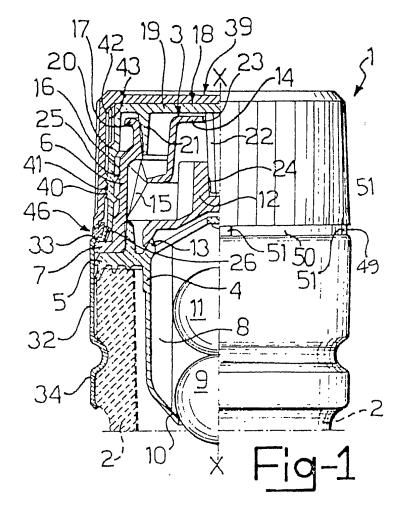
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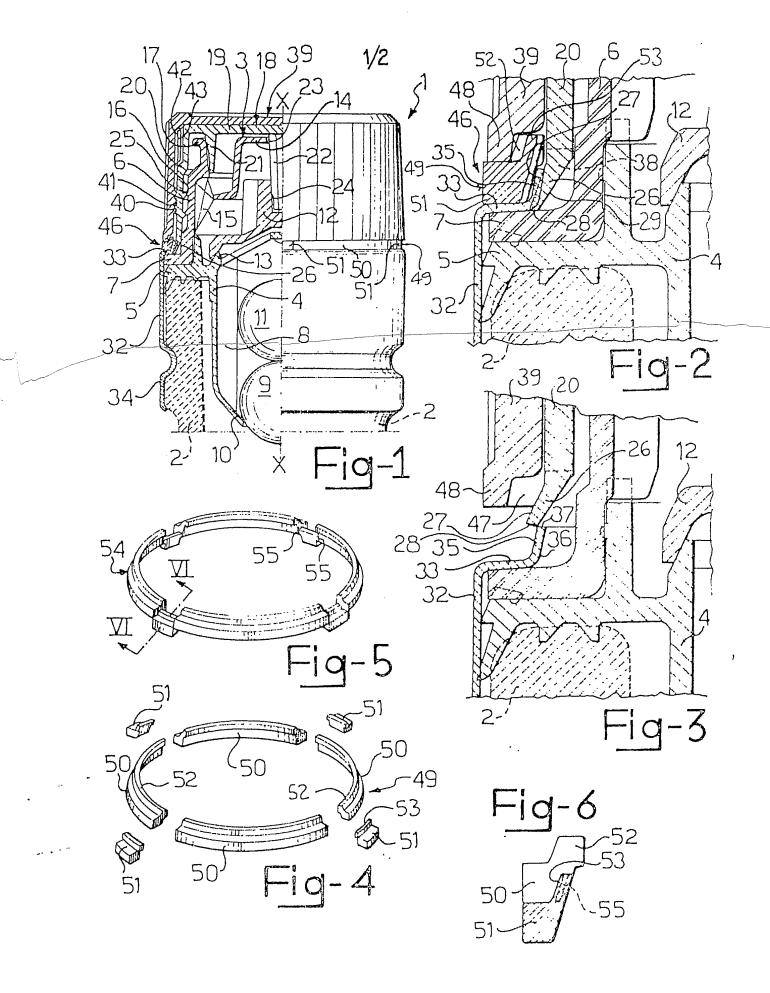
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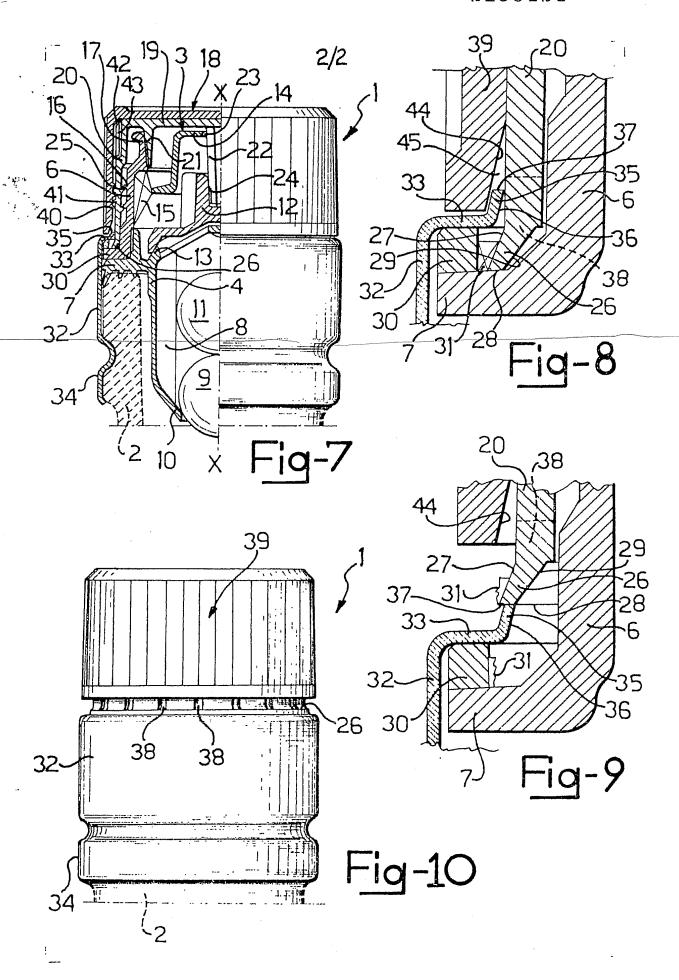
- (51) INT CL4 B65D 55/08
- (52) Domestic classification **B8T 13A TC**
- (56) Documents cited None
- (58) Field of search **B8T**

(54) Security closure for bottles

(57) A security closure (1) for bottles, adapted to render evident any fraudulent opening and reclosing, comprises a pourer (6), a stopper cap (18), an outer annular projection (26) projecting from the stopper cap (18) and having an inclined back, an external metal band (32) having a bent edge portion (33) for retaining the pourer (6) on the bottle (2), and an annular lip (35) projecting from the edge portion (33) and inclined upwardly and inwardly for cooperating with the projection (26).







SPECIFICATION

Security closure for bottles

5 The present invention relates to a security closure for bottles.

Such closures are used at present to render obvious the fact that a bottle has been opened and reclosed, possibly fraudulently.

In fact, such closures do not reassume their initial appearance when the stopper is screwed back on after its first unscrewing. Thus, a bottle which has been fraudulently opened can be distinguished from other intact
 bottles, for example from among those on sale in a supermarket.

One example of a closure of the type specified is that described in Italian Patent No. 793,760 of the present Applicants.

20 With this closure, when the stopper is screwed back onto the central body, it does not reach the initial axial position it had prior to the first unscrewing but is stopped before the engagement of the annular projection

25 against the ring. The configuration of this closure when the stopper is screwed back on is therefore only slightly different from the initial configuration, so that the occurrence of opening is not readily recognisable, particu-30 larly by a hasty purchaser.

The problem behind the present invention is that of providing a security closure for bottles which has structural and functional characteristics such that it presents a marked and more recognisable difference between the configuration of a violated closure and the configuration of an intact closure.

This problem is solved by a security closure including a pourer, a stopper cap, an outer 40 annular projection projecting from the stopper cap and having an inclined dorsal surface, an external metal band having a bent edge portion for retaining the pourer on the bottle, and an annular lip projecting from the edge portion and inclined approach to an annular lip projecting from the edge portion.

45 tion and inclined upwardly and inwardly for cooperating with the projection.

Further characteristics and advantages of the closure according to the present invention will become apparent from the following description of a preferred embodiment, given by way of non-limiting example with reference to the appended drawings, in which:

Figure 1 is a partially sectioned view of a closure according to the invention,

Figure 2 is a sectional view of a detail of the closure of Figure 1 on an enlarged scale,

Figure 3 is a view of the same detail as Figure 2 in a different position of operation of the closure,

60 Figure 4 is a perspective view of a detail of the closure of Figure 1, shown with the parts separated,

Figure 5 is a perspective view of the detail of Figure 4 as obtained by moulding,

Figure 5,

Figure 7 is a partially sectioned view of a variant of the embodiment of the closure according to the invention,

70 Figure 8 is a sectional view of a detail of the closure of Figure 7 on an enlarged scale,

Figure 9 is a view of a detail of Figure 8 in a different position of operation of the closure, and

75 Figure 10 is a view of the closure of Figure 7 in a different position of operation.

With reference to the appended drawings, there is generally indicated 1 a security closure for a bottle 2 of which there is shown in broken outline only its upwardly open neck portion having an axis X-X.

The closure 1 comprises a central body 3 which extends along the axis X-X and is formed by a first tubular member 4 partially 85 inserted in the neck of the bottle 2 and having a peripheral flange 5 bearing thereon, and by a second tubular member 6, termed a pourer, press fitted onto the first member 4 as an extension thereof and having a peripheral 90 flange 7 bearing against the flange 5.

The first member 4 and the second member 6 are made from suitable plastics materials, for example polythene and polystyrene respectively.

95 The first tubular member 4 is formed with an internal cage 8 for retaining and guiding along the axis X-X a first glass ball 9 which acts as a shutter for the central body 3 and is movable towards and away from a shutter 100 seat 10 formed in the first tubular member 4, as well as second glass ball 11 which acts as a striker for the first ball 9.

A bell shutter 12 is movable in the second tubular member 6 between a shutter seat 13 105 formed in the first tubular member 4 and a base stop 14 fixed to the second tubular member 6 by spokes 15.

The second tubular member 6 of the central body 3 has external screw threading 16 and 110 terminates with a drip-preventing lip 17.

The closure 1 also includes a stopper cap 18 having an end wall 19 and a downwardlyfacing cylindrical skirt 20 with an axis X-X. The stopper 18 is formed from a suitable 115 plastics material such as polypropylene.

A thin tubular appendage 21 with an axis X-X projects downwardly from the end wall 19 and is inserted with a fluid-tight seal in the drip-preventing lip 17, and a torsionally rigid 120 splined boss 22 with an axis X-X extends through a hole 23 in the base 14 into engagement with a grooved seat 24 in the bell shutter 12.

The cylindrical skirt 20 of the stopper cap 125 18 has internal screw threading 25 engaged with the threading 16 of the second tubular member 6.

An outer annular projection 26 projects from the cylindrical skirt 20 of the stopper

This projection 26 has a saw-toothed profile with an inclined dorsal surface 27 and a downwardly-facing frontal surface 28. To advantage, the stopper cap 18 has a bevel 29 formed internally on its downwardly-facing free edge. This bevel 29 gives the projection 26 a predetermined degree of resilient yielda-

The closure 1 further includes an external 10 metal band 32, for example of aluminium alloy, which is fitted around the flanges 7, 5 and the neck of the bottle 2.

The band 32 has an upper edge portion 33 bent horizontally ovef the flange 7 and a 15 lower portion 34 deformed into an S-shape to engage an S-profile on the neck of the bottle 2. The band 32 thus retains the pourer 6 of the central body 3 on the bottle 2.

The upper edge portion 33 has an annular 20 lip 35 inclined upwardly and inwardly, and located above the dorsal surface 27 of the projection 26.

The lip 35 has an inner surface 36 with an inclination substantially the same as the incli-25 nation of the dorsal surface 27, and a flat frontal apex 37.

At the free end of its skirt 20, the stopper cap 18 has a plurality of spaced-apart apertures, all indicated 38.

A stopper cover 39 is press-fitted onto the 30 stopper cap 18 and is formed of a suitable plastics material, for example polypropylene. To advantage, the polypropylene of the stopper cap 18 and that of the stopper cover 39 35 are of different colours so that there is a colour contrast between the stopper 18 and the stopper cover 39.

The stopper cover 39 is retained axially on the stopper 18 by the snap-engagement of 40 annular projections 40 and 41 formed respectively on the inside of the stopper cover and the outside of the stopper.

As regards the fixing of the stopper cover 39 to the stopper 18 for rotation therewith, 45 this is achieved by axial teeth 42 and 43 formed respectively on the inside of the stopper cover and the outside the stopper and engaged with each other by a splined coupling.

50 The stopper cover 39 is externally knurled in an entirely conventional manner and defines an annular seat 46 in correspondence with the projection 26 defined by the edge portion 33 and the lip 35. The annular seat 55 46 includes an annular recess 47 defined by an overhang 48 formed on the stopper cover 39.

The ring 49 is formed by a plurality of sectors of different angular extents. In particu-60 lar, it includes four sectors, each indicated 50, having a larger angular extent, for example 60°, and four sectors, each indicated 51, having a smaller angular extent, for example 30° disposed alternately.

Each sector 50 (51) is formed with an edge

52 (53) which is housed in the recess 47.

It should be noted that the edges 52 of the sectors 50 have sections of such a size as to be housed in the recess 47 with a small 70 clearance, substantially wedged between the overhang 48 and the skift 20.

The edges 53 of the sectors 51, however, have sections of such a size as to be housed in the recess 47 with a large clearance so as 75 to be loose between the overhang 48 and the skirt 20.

It should also be noted that the sectors 50 and 51 are made by moulding in a single, substantially annular piece, indicated 54, pre-80 ferably made from polystyrene.

The sectors 50 and 51 are axially offset from each other in the piece 54 and are interconnected by rupturable links, each indicated 55.

85 In particular, the sectors 50 and 51 are staggered so that the sectors 51 are connected to the sectors 50 in correspondence with the edges 53.

The piece 54 is arranged in advance of the 90 cap-shaped stopper cover 39. During fitting of the stopper cover 39 onto the stopper 18, the links 55 are broken and the sectors 50, 51 arrange themselves coplanarly so as to form the ring 49.

The operation of the closure 1 according to the invention is described below with reference to an initial condition illustrated in Figures 1 and 2, in which the closure is intact.

When the assembly constituted by the stop-100 per cover 39 and the stopper 18 is unscrewed, the projection snaps over the lip 35 and at the same time the distance between the stopper cover 39 and the edge 33 in-

105 Hence, on the one hand, the snap action produces a noise which indicates the correct unscrewing and, on the other hand, the annular seat 46 is opened. This opening causes the release of the sectors 50 and 51 which,

110 not being retained, fall from the closure. When the stopper is screwed back onto the central body, it no longer reaches the initial position described above but is stopped before this in a position shown in Figure 3, in

115 which the surface 28 of the projection 26 bears against the facing apex 37 of the annular lip 35.

Furthermore, it does not have the ring 49. This ring, in fact, cannot be put back into use 120 fraudulently due to the practical impossibility of recovering and assembling the sectors 50, 51 and holding them during the screwing up of the stopper. In particular, it is practically impossible to hold the sectors 51 which are 125 housed loosely in the seat 46.

In this position, the free end of the stopper 18 is clearly visible, this visiblity being accentuated by the presence of the apertures 38 in the stopper 18 and by the contrast in colour

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With reference to Figures 7, 8, 9 and 10, a closure according to a variant of the embodiment of the invention is described below.

In order not to overburden the description, the parts corresponding to those of the closure of Figures 1 to 6 have the same reference numerals and will not be described below.

According to this variant, the closure 1
10 includes a ring 30 of polypropylene which extends coaxially outside the stopper cap 18 at the level of and surrounding the dorsal surface 27 of the projection 26. The ring 30 is engaged with the dorsal surface 27 of the 15 projection 26 to which it is connected by a plurality of rupturable radial links, each indicated 31, and bears on the flange 7 of the second member 6 of the central body 3.

The band 32 is fitted onto the ring 30 and 20 is bent-horizontally over the edge portion 33.

Thus, the band 32 retains the ring 30 and hence the stopper cap 18 on the bottle 2.

The stopper cover 39 extends over the stopper 18 as far as the apertures 38 and has 25 an internal bevel 44 which defines a seat 45 for the annular lip 35.

The operation of the closure 1 according to this variant is described below with reference to an initial condition illustrated in Figures 7 30 and 8, in which the closure is intact.

As the unscrewing of the assembly constituted by the stopper cover 39 and the stopper 18 is started, the rupturable links 31 are broken and the projection 26 is therefore 35 separated and removed from the ring 30. This ring 30 is in fact retained on the pourer body 3 and the bottle 2 by the presence of the band 32.

As the unscrewing is continued, the projec-40 tion 26 engages and snaps over the annular lip 35. In particular, this engagement occurs between the dorsal surface 27 of the projection 26 and the inner surface 36 of the annular lip 35.

45 This snapping over is accompanied by a sharp noise which indicates the correct unscrewing of the stopper. This unscrewing is continued until the stopper itself is removed completely.

When the stopper is screwed back onto the central body, it no longer reaches the initial position described above but is stopped prior to this in a position shown in Figures 9 and 10, in which the surface 28 of the projection
bears against the facing apex 37 of the annular lip 35.

In this position, the free end of the stopper 18 is clearly visible, this visibility being accentuated by the presence of the apertures 38 in 60 the stopper 18 and by the contrast in colour between the stopper cover and the stopper.

The closure according to the invention renders the occurrence of the opening and reclosing of the bottle considerably more recognisa-

CLAIMS

Security closure for bottles, comprising a pourer, a stopper cap, an outer annular projection projecting from the stopper cap and having an inclined dorsal surface, an external metal band having a bent edge portion for retaining the pourer on the bottle, and an annular lip projecting from the edge portion and inclined upwardly and inwardly for cooperating with the projection.

Security closure according to Claim 1, in which the annular inclined lip has an inclination substantially the same as the inclination of the dorsal surface of the annular projection.

Security closure according to Claim 2, including a cap-shaped stopper cover pressfitted onto the outside of the stopper cap and defining an annular seat in correspondence
 with the projection, and a ring housed in the annular seat.

4. Security closure according to Claim 3, in which the ring is formed by a plurality of sectors.

90 5. Security closure according to Claim 4, in which the sectors of the ring are housed in the seat with large and small clearances alternately.

Security closure according to Claim 5, in
 which the sectors of the ring are connected together by rupturable links.

 Security closure according to Claim 2, including a ring located between the pourer and the edge portion and connected to the 100 annular projection by rupturable links.

8. Security closure according to Claim 2, including a plurality of apertures formed in the stopper cap in correspondence with the annular projection and spaced therearound.

9. Security closure according to Claim 8, including a cap-shaped stopper cover which is press-fitted onto the outside of the stopper cap as far as the apertures and is in colour contrast with the stopper cap.

110 10. Security closure according to Claim 2, in which the projection has a predetermined degree of resilient yieldability so as to snap over the annular lip with the emission of a noise.

115 11. Security closure for bottles, substantially as herein described with reference to, and as shown in, the accompanying drawings.

Printed in the United Kingdom for Her Majesty's Stationery Office, Dd 8818935, 1985, 4235. Published at The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

UK Patent Application (19) GB (11) 2 172 803 A

(43) Application published 1 Oct 198

(21) Application No 8607042 (22) Date of filing 21 Mar 1986 (30) Priority data (31) 8508173 (32) 28 Mar 1985 (33) GB	(51) INT CL ⁴ A61K 9/22 (52) Domestic classification (Edition H) A5B 829 834 836 L N (56) Documents cited GB A 2079152 GB A 2077585 GB A 2077586 EP A1 0042219
(71) Applicant STC plc (United Kingdom), 190 Strand, London WC2R 1DU	(58) Field of search A5B Selected US specifications from IPC sub-class A61K
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(54) Controlled delivery device

(57) A device for the controlled release of an active material into an aqueous medium, e.g. body fluids, comprises a water soluble glass container in which the active material is sealed. The container wall has a region of reduced thickness or made of glass of higher dissolution rate which region dissolves before the remainder of the container wall to release the active material.

Fig.1a.

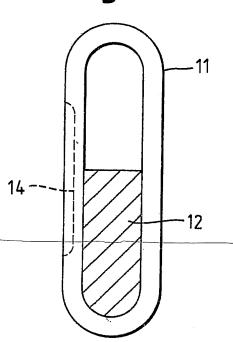


Fig. 1b.

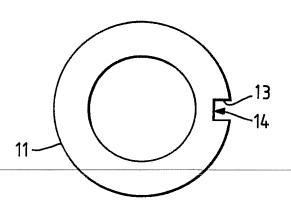


Fig.2.

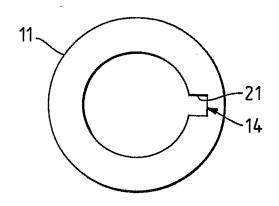


Fig. 3.

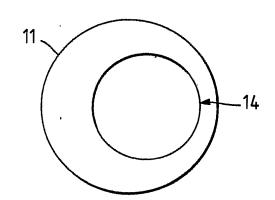


Fig.4.

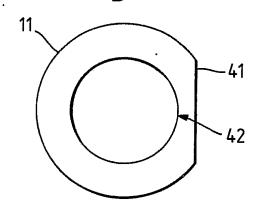
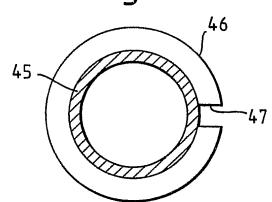
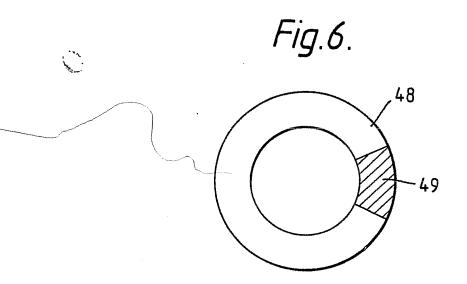
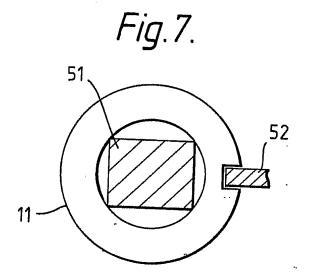


Fig. 5.







SPECIFICATION Controlled delivery device

This invention relates to devices for the controlled 5 release of an active material into an aqueous medium.

Active materials can be released, after a predetermined delay, into an aqueous medium by sealing the material in a tube of a water soluble glass. On exposure to the aqueous medium the glass dissolves at a predetermined rate and releases its contents when dissolution of the tube wall has taken place. Such a technique is described for example in our published specification No. 15 2,079,152 B.

A disadvantage of this technique is the difficulty of accurate prediction of the period before release is effected. This period is of course proportioned to the tube wall thickness but is affected by a variety of factors. The most important of these are nonconcentricity of the tube and the extreme fragility of the tube shortly before complete dissolution, i.e. the tube may collapse to release its contents before dissolution is complete. Both these factors reduce the expected delay period by an unpredictable amount

The object of the present invention is to minimise or to overcome this disadvantage.

According to one aspect of the invention there is provided a device for the release of an active material into an aqueous medium, the device including a sealed container of a water soluble glass in which the active material is encapsulated, said container being so constructed that, on exposure to water, a portion only of the container is selectively breached by dissolution to release the container contents after a predetermined period.

According to the invention there is further provided a device for the release of an active
40 material into an aqueous medium, the device including a sealed container of a water soluble glass in which the active material is encapsulated, wherein said container has a region of reduced resistance to solution attack whereby, in use, the
45 release time of the active material is determined.

By providing the capsule with a weak point, dissolution of a portion of the wall to release the contents is effected whilst the remainder of the wall remains self supporting. This ensures that the release time can be accurately predetermined.

Embodiments of the invention will now be described with reference to the accompanying drawings, in which:—

Figs. 1a and 1b are respectively cross-sectional and longitudinal sectional views of a capsule for release of an active material;

Figs. 2, 3, 4, 5 and 6 are sectional views of alternative capsules;

and Fig. 7 illustrates a method of making the capsule of Figs. 1a and 1b.

Referring to Figs. 1a and 1b, the controlled release device comprises a tubular capsule 11 formed from a water soluble glass composition and containing a quantity of an active material 12. The wall of the capsule is provided a growth a longitudinal growth.

13 whereby a region 14 of reduced wall thickness, i.e. of reduced resistance to solution attack, is provided. The dissolution time of the capsule wall is determined both by the thickness of the region 14
70 and the dissolution rate of the glass from which the capsule is made. When the capsule is exposed to an aqueous medium the whole capsule wall is dissolved at a constant rate. After a predetermined period the region 14 is completely dissolved thus
75 releasing the capsule contents. As only this small portion of the capsule wall is dissolved away the remainder of the capsule remains self supporting.

An alternative device construction is shown in Fig. 2. In this arrangement a groove 21 is provided on the internal wall of the capsule. A further technique is shown in Fig. 3, wherein a considerable degree of non-concentivity between the internal and external surfaces of the tube bore is provided. This determines a region of minimum wall thickness which in turn determines the dissolution time of the device. Another construction is shown in Fig. 4. The wall 11 of the capsule is cut away to form a flat portion 41 defining a reduced thickness region 42 whose thickness determines the dissolution time.

In a further application (Fig. 5) the capsule wall may comprise an inner layer 45 of a glass of relatively high dissolution rate and an outer layer 46 of a glass of relatively low dissolution rate. A region of reduced wall thickness, e.g. a groove or
 depression 47, extends substantially or completely through the outer layer 46 so that dissolution through the inner layer 45 can take place whilst the

outer layer remains self supporting.

A further embodiment is shown in Fig. 6. In this

device the wall of the capsule comprises a first glass
48 of relatively low dissolution rate and a second
glass 49, forming a sector of the wall, of a relatively
high dissolution rate. When the device is exposed to
an aqueous medium the sector comprising the high

105 solubility glass 49 dissolves preferentially to release the capsule contents.

A technique for forming the groove of the device of Fig. 1 is shown in Fig. 6. The tube 11, prior to filling and sealing, is mounted on a square section mandrel 41 on which the tube is a sliding fit. The tube 11 is held by the mandrel 51 against a cutting wheel 52 whereby the groove is formed. The depth of the groove is determined by the distance between the mould 51 and the wheel 52 thus providing a high degree of accuracy.

In an alternative fabrication process the tubes of Figs 1 to 6 may be formed by drawing from a cast or moulded preform, the cross-section of the tube corresponding to the preform cross-section. Such techniques are well known to those skilled in the glass processing art.

A wide variety of water soluble glass compositions may be used. However, for medical or veterinary applications we prefer to employ phosphorus pentoxide/alkali metal glasses modified by the addition of calcium oxide, magnesium oxide, zinc oxide or mixtures thereof. The glass may also contain trace quantities of therapeutic elements in

oxide form.

medicament for release into the body fluids of the host into which it is implanted or ingested.

The following example illustrates the invention:— A water soluble glass was prepared by fusion of a 5 mixture of sodium oxide, magnesium oxide, calcium oxide and phosphorus pentoxide to form a homogeneous material. Analysis of the glass showed its composition to be

10	Na ₂ O	40.3 mole percent
	MgO	8.8 mole percent
	CaO	7.8 mole percent
	$P_{2}O_{5}$	43.0 mole percent

15 The glass was cast into eccentric bore cylindrical tube stock having an outside diameter of 27 mm, an inner diameter of 15.5 mm. The bore was offset to provide a thinnest wall thickness of 2.6 mm. Portions of this stock were drawn down the tubing

20 having an outer diameter of 2 mm and a wall thickness of 0.2 mm at its thinnest point.

The drawn tube was cut into 10 mm sections, each of which was filled with methylene blue powder and sealed at both ends. The sealed tubes 25 were immersed in distilled water of pH 6.5 at 38°C and the time taken (T_d) for dissolution of the thin portion of the wall to release the dye was measured.

From a sample of 20 tubes the average dissolution 60 time T_d was found to be 49.4 h with a standard 30 deviation of 1.1 h or 2.2%. This demonstrates the close accuracy with which the dissolution time may be predetermined.

CLAIMS

1. A device for the release of an active material into an aqueous medium, the device including a sealed container of a water soluble glass in which the active material is encapsulated, said container being so constructed that, on exposure to water, a portion only of the container is selectively breached by dissolution to release the container contents after 40 a predetermined period.

2. A device for the release of an active material into an aqueous medium, the device including a sealed container of a water soluble glass in which the active material is encapsulated, wherein said container has a region of reduced resistance to solution attack whereby, in use, the release time of the active material is determined.

3. A device as claimed in claim 2, wherein the reduced resistance region comprises a region of reduced wall thickness.

4. A device as claimed in claim 3, wherein said reduced thickness region comprises a groove provided in the device wall.

5. A device as claimed in any one of claims 1 to 4, wherein the glass is a phosphorus pentoxide glass.

6. A device as claimed in claim 5, wherein said glass includes calcium oxide, zinc oxide or mixtures thereof.

7. A device for the release of an active material into an aqueous medium substantially as described herein with reference to Fig. 1, 2, 3 or 4 of the accompanying drawings.

Printed for Her Majesty's Stationery Office by Courier Press, Leamington Spa, 10/1986. Demand No. 8817356. Published by the Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.

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UK Patent Application (19) GB (11) 2 264 110 (13) A

(43) Date of A publication 18.08.1993

(21) Application No 9202448.8

(22) Date of filing 05.02.1992

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(51) INT CL5 B65D 47/28 41/32

(52) UK CL (Edition L) B8T TWH T14D U1S S1808 S1825

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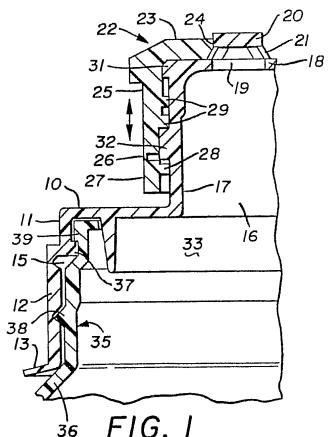
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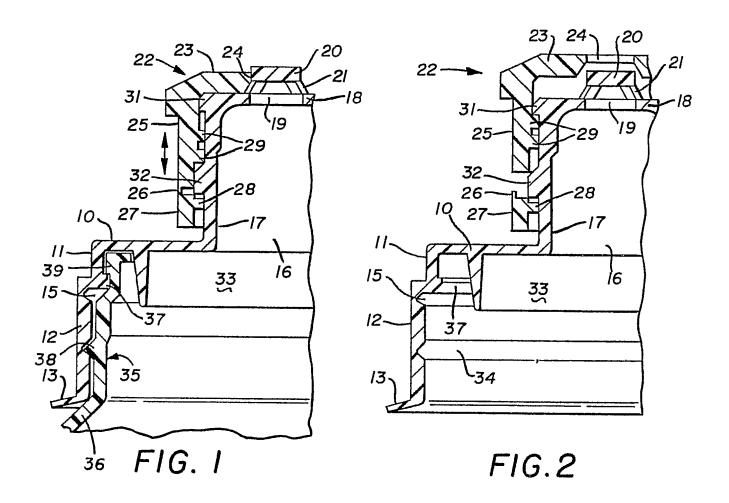
(58) Field of search

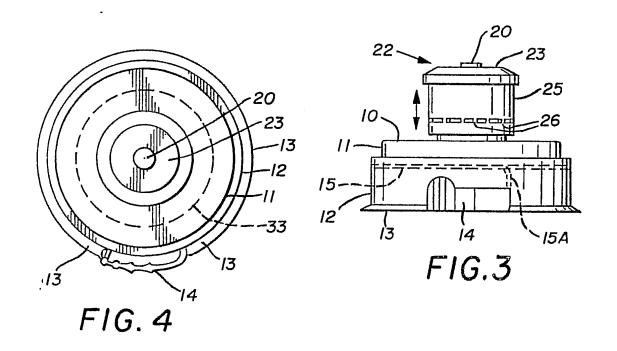
UK CL (Edition K) B8T TTB TTC TWH INT CL5 B65D 41/32 47/26 47/28

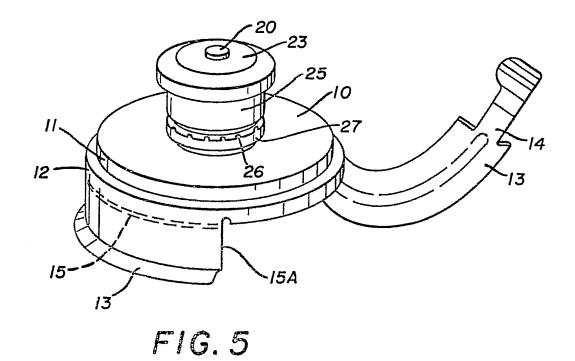
(54) Resealable bottle cap with push-pull closure

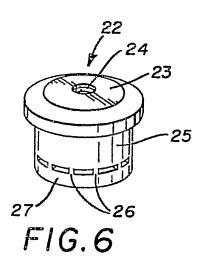
(57) A tamper-evident double cap for a bottle, such as a sport's water bottle, comprises a main cap attached to the bottle 36 and having an annular tear skirt 13 with a pull tab and a tear groove (14, 15 Figure 3), an upstanding pour spout 17 registering with an opening 16 in the cap and a plug 20 positioned thereabove on a plurality of legs 21 on the pour spout. Annular ribs 31, 32 are formed on the exterior of the pour spout to permit reciprocating movement of a push pull top cap 22 having an opening 24 therein registrable with the plug 20. A tamper evident annular ring 27 of the top cap has an internal annular flange 28 positioned on the pour spout below one of said annular ribs 32 and is detachably secured to the top cap by a plurality of frangible elements (26) which break on first upward movement of the top cap. The main cap, together with the top cap, is replaceable on the water bottle when the annular tear skirt is removed, annular flange 37 engaging a groove in the bottle neck.











RESEALABLE BOTTLE CAP WITH PUSH-PULL CLOSURE

This invention relates to a resealable bottle cap with a push-pull closure and in particular to tamper indicating closures for containers, such as blow moulded plastics bottles, such as used by sport's figures as water bottles and/or beverage containers.

Prior closures of this type may be seen in U.S. Patent Nos. 3,902,621, 4,469,253, 4,500,016, 4,561,553, 4,589,561, 4,801,032 and 4,948,003.

The present cap in part resembles the tamper-evident closure of U.S. patent 4,589,561.

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According to the present invention, there is provided a resilient, resealable bottle cap for a bottle having a neck with dual bottle cap retaining means on the exterior of the neck, said cap having an annular flange depending from a cap top for surrounding the bottle neck, said flange having means for engaging said cap retaining means, and internal annular groove, the depending flange below said groove defining a tear skirt with a pull tab; there being a pour spout on said top communicating with an opening in said top, said pour spout having an apertured secondary top thereon and means on said secondary top positioning a plug in spaced relation to said secondary top and its aperture, and there being a top cap movably disposed on said spout and having an opening registering with said plug to form a closure when said top cap is in a first position, said top cap including a ring and a plurality of frangible elements integrally connecting said ring with another part of said top cap in said first position; and a flange on said spout retaining said ring in said first position when said top cap is moved away from said first position to a second

position to locate said opening in said top cap in spaced relation to said plug.

The invention also extends to a combination of the cap and a bottle on which it is fitted.

For a better understanding of the invention and to show how the same may be carried into effect, reference will now be made, by way of example, to the accompanying drawings, in which:-

Figure 1 is a vertical section through a portion of a resealable bottle cap with a push-pull top portion thereof in closed and sealed position;

Figure 2 is a view similar to Figure 1 with the push-pull top portion shown in open position;

Figure 3 is a side view of the bottle cap;

Figure 4 is a plan view of the bottle cap;

Figure 5 is a perspective view of the bottle cap with a portion of a tear skirt removed; and

Figure 6 is a perspective view of a top cap portion of the cap prior to assembly on the remainder of the cap.

Referring firstly to Figure 5, a resealable bottle cap has a push-pull closure positioned thereon and includes a top portion 10 with a depending annular flange of different diameters, one of which forms a relatively short first portion 11 and the other forms a larger diameter portion 12.

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An out-turned flange 13 is formed over most of the lower edge of the portion 12 and a pull tab 14 is provided on the portion 12 in the area between the ends of the out-turned flange 13.

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The portion 12 is provided with an annular area of weakness comprising an internal tear groove 15 which enables the portion 12 to be removed when the tear tab 14 is held and moved outwardly and around the resealable bottle cap.

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In Figure 1, it will be seen that an opening 16 is formed centrally of the top 10 of the cap and that an upstanding cylindrical pour spout 17 is positioned in registry with the opening 16, the upper end of the spout 17 having a secondary top portion 18 thereon which is apertured at 19. A plug 20 is positioned on the secondary top 18 in spaced relation to the aperture 19 by a plurality of circumferentially-spaced, angularly arranged legs 21.

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A push-pull top cap 22 is positioned on the spout 17 and comprises a top portion 23 having a central opening 24 therein which registers with the plug 20 to form a closure when the top portion 23 is in the position illustrated in Figure 1 and resting on the secondary top 18 of the spout 17. The top cap 22 has a depending cylindrical body 25 and is connected by a plurality of frangible elements 26 on its lower edge with a ring 27. The ring 27 has an inturned annular flange 28 thereon which slidably engages with the exterior of the pour spout 17. The body 25 of the top cap 22 has two inturned annular flanges 29 which also slidably engage the outer surface of the pour spout 17. two annular flanges 31 and 32 respectively, formed on the exterior of the pour spout 17, the flange 31 being oppositely disposed with respect to the secondary top 18 and the flange 32 being spaced therebelow and above the top

10 of the bottle cap so that reciprocating movement of the push-pull top cap 22 is limited.

In the assembled form illustrated in Figure 1, the integral joined by the frangible elements 26 cylindrical body 25 of the top cap 22 is incapable of moving upwardly due to the inter-engagement of the flange 28 thereon with the flange 32 on the spout 17, whilst the cylindrical body 25 of the top cap 22 is incapable of vertical movement such as necessary to move the apertured top 23 thereof above the plug 20 until sufficient force is applied to the top cap 22 to break the frangible elements 26, whereby the top cap 22 can move to the position illustrated in Figure 2 wherein the opening 24 therein moves upwardly and away from the plug 20. The flanges 29 on the cylindrical body 25 of the top cap 22 cannot move above the flange 31 on the secondary top 18 of the spout 17 so that the push-pull top cap of the bottle cap is childsafe as it is not removable therefrom.

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Figure 3 shows the resealable bottle cap in its initial assembled condition and it will be seen that there is an area of weakness 15A extending between the lower edge of the larger portion 12 and the tear groove 15.

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The portion 12 of the depending flange engages a neck finish 35 of a water bottle 36 (Figure 1) or the like and is provided with two configurations for engaging, matching or registering with configurations on the neck finish. These comprise an internal annular flange 37 on the lower inner edge of the flange portion 11 of the bottle cap, which is in opposed relation to the conventional depending sealing flange 33 of such caps, and an annular groove 34 in the larger diameter flange portion 12. The blow moulded water bottle 36 or similar containers having an

appropriately shaped neck finish 35 including annular ribs 38 and 39 on their outer surfaces will accordingly register with the flange 37 and the groove 34 of the present cap.

It will thus be seen that the present resealable bottle cap with a push-pull closure forms an attractive and very practical closure for a water bottle such as used in various sports such as bicycle races and the like, in that the bottles which are initially filled and sealed by the application of the resealable bottle cap will retain the due to the registering position in configurations in the depending annular flanges of the portion of the cap engaging the water bottle finish and at the same time provide a tamper-indicating sealed closure with respect to the upstanding cylindrical pour spout which must be forcibly opened by moving the push-pull top cap 22 upwardly so as to break the frangible elements 26 to open the aperture 19 therein and permit the contents of the water bottle to be used. At such time as the water bottle 36 is empty, it may be refilled by using the tear tab 14 to remove the major portion of the larger flange portion 12 from the bottle cap, which can then be removed by forcibly disengaging the inturned annular flange 37 from a matching configuration on the water bottle neck finish. This latter feature permits the bottle cap to be repositioned on the refilled water bottle where it holds tightly and permits the push-pull top cap 22 to be opened and closed in the same manner as was provided by the original complete resealable bottle cap assembly.

The cap may be formed of a resilient, moulded plastics material such as polyethylene.

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CLAIMS:

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- A resilient, resealable bottle cap for a bottle having a neck with dual bottle cap retaining means on the exterior of the neck, said cap having an annular flange depending from a cap top for surrounding the bottle neck, said flange having means for engaging said cap retaining means, and an internal annular groove, the depending flange below said groove defining a tear skirt with a pull tab; there being a pour spout on said top communicating with an opening in said top, said pour spout having an apertured secondary top thereon and means on said secondary top positioning a plug in spaced relation to said secondary top and its aperture, and there being a top cap movably disposed on said spout and having an opening registering with said plug to form a closure when said top cap is in a first position, said top cap including a ring and a plurality of frangible elements integrally connecting said ring with another part of said top cap in said first position; and a flange on said spout retaining said ring in said first position when said top cap is moved away from said first position to a second position to locate said opening in said top cap in spaced relation to said plug.
- 2. A cap according to claim 1, wherein said means on said pour spout and on said ring retaining said ring in said first position comprise an annular flange on said pour spout and an inturned annular flange on said ring.
- 3. A cap according to claim 1 or 2, wherein said pour spout has a tubular body and said top cap has a cylindrical body, the inner diameter of which is greater than the outer diameter of said tubular body.

- 4. A cap according to claim 1, 2 or 3, wherein said means on said secondary top spacing said plug with respect to said secondary top consists of upstanding legs integrally formed with said secondary top and said plug, said legs being registrable with said top portion when in said first position.
- 5. A cap according to any one of the preceding claims and being made of moulded plastics material.
- 6. A resilient, resealable bottle cap, substantially as hereinbefore described with reference to the accompanying drawings.

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- 7. A combination of a resilient, resealable bottle cap according to any one of the preceding claims fitted on a neck of a bottle having dual bottle cap retaining means on the exterior of said neck.
- 8. A combination according to claim 7, wherein said dual retaining means comprises annular flanges on said neck, and said means on said depending flange for engaging said dual retaining means comprises an inner annular flange and groove.
 - 9. A combination according to claim 7 or 8, substantially as hereinbefore described.

Amendments to the claims have been filed as follows

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- A resilient, resealable bottle cap for a bottle having 1. a neck with bottle cap retaining means on the exterior of the neck, said cap having an annular flange depending from a cap top for surrounding the bottle neck, said flange having means for engaging said cap retaining means, and an internal annular groove, the depending flange below said groove defining a tear skirt with a pull tab; there being a cylindrical pour spout on said top communicating with an opening in said top, said pour spout having an apertured secondary top thereon and means on said secondary top positioning a plug in spaced relation to said secondary top and its aperture, and there being a cylindrical top cap rotatably and axially movably disposed on said spout and having an opening registering with said plug to form a closure when said top cap is in a first position, said top cap including a ring and a plurality of circumferentially spaced frangible elements integrally connecting said ring with a lower edge of said top cap in said first position; and an inturned flange on said spout retaining said ring in said first position when said top cap is moved axially away from said first position so as to break said frangible elements and move said top cap to a second position to locate said opening in said top cap in spaced relation to said plug.
- 2. A cap according to claim 1, wherein an out-turned annular flange on said pour spout and said inturned annular flange on said ring act to retain said ring in said first position.
- 3. A cap according to claim 1 or 2, wherein said pour spout has a tubular body and said top cap has a cylindrical body, the inner diameter of which is greater than the outer diameter of said tubular body.

9202448.8

Relevant Technical fi	elds			Search Examiner
(i) UK CI (Edition	K)	B8T (TWH; TTC; TTB)	Goardin Examinor
(ii) Int CL (Edition	5)	B65D 47/26, 47/28, 41/32	L HARDEN
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(i) UK Patent Office				Date of Search 5 MAY 1992

category see over)	Identity of document and relevant passages						
У	GB 2242678 A (STULL) See figure 2	1-5 7 and 8					
Y	GB 2200345 A (STULL) See figure 2	1, 3, 4 5, 7 ar					
Y	GB 2172275 A (STULL) See figure 4	1-5 7 and 8					
У	EP 0244327 A1 (SOCIETE NOUVELLE) See figure 6	1-5, 7 and 8					
У	US 4589561 (CRISCI) See figure 1	1-5, 7 and 8					
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(11) **EP 1 101 707 A1**

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication: 23.05.2001 Bulletin 2001/21

(51) Int CI.7: **B65D 51/20**, B65D 43/02

(21) Application number: 00310164.9

(22) Date of filing: 15.11.2000

(84) Designated Contracting States:

AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU

MC NL PT SE TR

Designated Extension States:

AL LT LV MK RO SI

(30) Priority: 15.11.1999 US 440383

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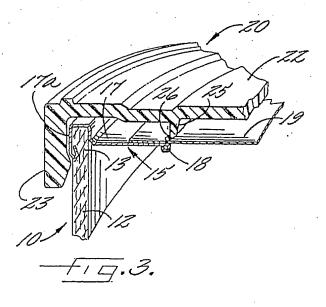
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(54) Resealing overcap for a cylindrical container

(57) A resealing overcap (20) is provided for fitting over a top end (13) of a cylindrical container (10) and an easy-open top end closure (15) thereon which includes an end ring (17) having an inside circular downwardly and inwardly curved bead (18) defining a central opening and closed by a removable membrane (19) positioned thereon. The resealing overcap (20) includes a circular central portion (22) having an outer periphery and adapted to extend radially of the container when positioned thereon for covering the top end closure (15). The overcap (20) further includes a downwardly depending flange (23) integrally extending from and around the central portion outer peripheral area and adapted to extend axially of the cylindrical container

when positioned thereon to provide a friction fit around the end ring (17) of the container end closure when positioned thereon. The resealing overcap (20) further includes a downwardly depending flexible ring portion (25) integrally extending from an inside surface of the overcap central portion (22) and adapted to extend axially of the cylindrical container when positioned thereon to define a leading edge portion (26) adapted to be positioned over the inside circular bead (18) of the container end closure when positioned thereon before opening of the container and adapted to flex and snap fit around the bead (18) of the end ring of the container end closure upon opening of the container and removal of the easy-open membrane patch for resealing of the container.



Description

Field Of The Invention

[0001] This invention relates to an improved construction of an overcap for fitting over a top end of a cylindrical container and an easy-open top end closure thereon which includes an end ring defining a central opening closed by a removable membrane.

Background Of The Invention

[0002] Easy-open containers have heretofore been provided for a variety of products including powdered products, such as food products, cleaning products, etc. These easy-open containers are often constructed of a composite cylindrical body portion having end closures for closing and sealing the container and wherein the top end closure comprises an end ring seamed to the composite container body and having an inside circular peripheral area in the form of a downwardly and inwardly curved bead and defining a central opening of desired size to have access therethrough to the interior of the container. The top end closure further includes an easyopen membrane patch of sufficient size to cover the central opening and attached to the end ring for allowing detachment and removal when it is desired to open the container to obtain access to the product therein.

[0003] These types of easy-open containers often also include overcaps constructed for fitting over the container top end portion and top end closure before and after opening of the container. These overcaps serve many functions including, but not limited to, protecting the top of the container from damage before and after removal of the membrane and opening of the container, keeping unwanted items from getting into the container after removal of the membrane and opening of the container, keeping the product within the container from spilling out of the container after opening of the container, helping improve stack-ability of these containers before and after opening, and increasing the life of the product after opening of the container.

[0004] With the increased packaging of moisture and oxygen sensitive products into these types of containers, the need has been established for an improved reseal feature for the container, after opening of the container and removal of the membrane, to re-seal the container and prevent undesirable moisture and oxygen from entering the container.

Object And Summary Of The Invention

[0005] Accordingly, it is the object of this invention to provide a generally circular overcap adapted to fit over a top end of a cylindrical container and top end closure thereon which includes an end ring having an inside circular downwardly and inwardly curved bead defining a central opening and closed by a removable membrane

positioned thereon, wherein the overcap construction includes a resealing feature for resealing of the container after initial opening thereof and removal of the membrane from the top end closure.

[0006] It has been found by this invention that this object may be accomplished by providing a resealing overcap comprising a circular central portion having an outer periphery and adapted to extend radially of the container when positioned thereon for covering the container top end portion. The overcap further includes a downwardly depending flange portion integrally extending from and around the central portion outer peripheral area and adapted to extend axially of the cylindrical container when positioned thereon and having an inside diameter approximately equal to the outside diameter of the cylindrical container to provide a friction fit around the end ring of the container when positioned thereon.

[0007] The overcap further includes a downwardly depending flexible ring portion integrally extending from an inside surface of the overcap central portion and adapted to extend axially of the cylindrical container when positioned thereon to define a leading edge portion adapted to be positioned over the inside circular bead of the container end closure when positioned thereon before opening of the container and adapted to flex and snap fit around the downwardly and inwardly curved bead of the end ring of the container end closure upon opening of the container and removal of the easy-open membrane patch for resealing of the container. It has been found preferable to have the leading edge of the flexible ring of the overcap of a diameter smaller than the central opening of the container top end closure and to extend outwardly of the overcap at a predetermined angle of between 30° and 45° to the overcap downwardly depending flexible ring. It has also been found preferable to construct the overcap as an injection molded plastic overcap.

Brief Description Of The Drawings

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[0008] An object and advantages of this invention have been set forth above, other objects and advantages of the invention will become apparent in the detailed description of a preferred embodiment of this invention to be described below, when taken in conjunction with the accompanying drawings, in which:

FIG. 1 is a perspective view of an easy-open container having the improved overcap of this invention thereon and showing the overcap in exploded position;

FIG. 2 is a perspective view of the top portion of the container of FIG. 1 with the top end closure thereon and illustrating the membrane of such top end closure being removed for opening of the container to obtain access to the product therein;

FIG. 3 is a sectional view of one-side of the top portion of the container of FIG. 1, before removal of the

membrane of the top end closure, and with the overcap in position thereon; and

FIG. 4 is a view, like FIG. 3, after the membrane has been removed from the top end closure and the overcap repositioned on the end of the container and snap-fitted into the opening in the end closure formed by removal of the membrane for resealing of the container.

Detailed Description Of Preferred Embodiment Of The Invention

[0009] Referring now to the drawings, there is shown in FIG. 1 an easy-open container, generally indicated at 10, which is adapted for being filled with a powdered product P, such as food products, cleaning products, etc. The container 10 may be of any desired configuration and may be constructed of any desired material including composites, plastic, metal, etc. However, it is preferred that the container 10 be of a generally cylindrical configuration and be constructed of composite materials including paper layers of the type normally utilized for constructing containers for powdered products P and which are well understood by those with ordinary skill in the art.

[0010] The container 10 comprises a generally cylindrical container body 12 defining top and bottom opposite end portions 13, 14. The container 10 further includes top and bottom end closures 15, 16 attached respectively to the top and bottom container body end portions 13, 14 for closing and sealing the container with product P therein in a manner well understood by those with ordinary skill in the art. The bottom end closurel 6 may be any suitable end closure attached to or seamed to the bottom end portion 14 of the container and may be constructed of metal, composites, plastic or other suitable material.

[0011] The top end closure 15 is of an easy-open type which comprises an end ring 17 having an outside circular peripheral area 17a seamed to the container body top end portion 13 (as shown particularly in FIGS. 3 and 4). The end ring 17 further includes an inside circular peripheral area in the form of a downwardly and inwardly curved bead 18 which defines a central opening of desired size to allow access therethrough to the interior of the container 10 (as shown particularly in FIG. 2). The top end closure 15 further includes an easy-open membrane patch 19 of sufficient size to cover the central opening and which is attached to the end ring 17 for allowing detachment and removal of the membrane patch. 19 (as shown in FIG. 2) when it is desired to open the container 10 to obtain access to the product P therein. [0012] The end ring 17 of the top end closure 15 may be constructed of any suitable material including composites, metal, plastic, etc. and the membrane patch 55 may be constructed of any suitable material including composites, metal, plastic, etc. The bond or attachment between the membrane patch 19 and the end ring 17

may be any suitable bond including heat seal, adhesive etc. to create a bond of sufficient strength to resist internal forces within the container, while being sufficiently weak in the tensile direction to allow sealing of the bond for easy-opening of the top end closure 15 of the container 10, in a manner well understood by those with ordinary skill in the art. Preferably the bond may be formed with a polypropylene heat seal layer on the membrane patch or the end ring or both.

[0013] In accordance with this invention, a resealing overcap 20 is constructed for fitting over the container body top end portion 13 and the top end closure 15 before and after opening of the container 10 (as shown particularly in FIGS. 3 and 4) and for snap fitting into the central opening of the top end closure 15 after opening of the container 10 and removal of the easy-open membrane patch 19 for resealing of the container (as shown in FIG. 4).

[0014] The overcap 20 comprises a circular central portion 22 having an outer periphery and extending radially of the container 10 for covering the container top end portion 15. The overcap 20 further includes a downwardly depending flange portion 23 which integrally extends from and around the outer peripheral area of the central portion 22 and axially of the container body 12 when positioned on the container 10. This flange portion 23 has an inside diameter approximately equal to an outside diameter of the container body 12 to provide a friction fit around the end ring 17 and the outside peripheral area 17a thereof which is seamed to the top container body end portion 13 (as shown in FIGS. 3 and 4). [0015] The resealing overcap 20 further includes a downwardly depending flexible ring portion 25 integrally extending from an inside surface of the overcap central portion 22 and axially of the container body 12 when positioned on the container 10 to define a leading edge portion 26. This leading edge portion 26 is positioned over the inside peripheral area or bead 18 of the top end closure end ring 17 before opening of the container 10 (as shown in FIG. 3) and is adapted to flex and snap fit around the downwardly and inwardly curved bead 18 on the inside peripheral area of the top end closure 15 upon opening of the container 10 and removal of the easyopen membrane patch 19 (as shown in FIG. 4). This may be accomplished (as shown in FIG. 4) by exerting pressure with a finger or the like on the middle portion of the central portion 22 of the resealing overcap 20 which causes the leading edge portion 26 of the downwardly depending flexible ring portion 25 to flex and snap fit around the bead 18 on the inside of the end ring 17 of the end closure 15 of the container 10.

[0016] As may be seen in FIGS. 3 and 4, the leading edge portion 26 of the downwardly depending ring 25 of the resealing overcap 20 is preferably of a diameter larger than a diameter of the inside peripheral area defined by the bead 26 of the end ring 17 of the top end closure 15 of the container 10 and extends outwardly of the overcap 20 at a predetermined angle 6 to the downward-

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ly depending ring 25 thereof so as to effect the flexing and snap-fitting around the downwardly and inwardly curved bead 26 of the end closure 15 of the container 10 when re-sealing is desired. It is preferable that the predetermined angle θ of the leading edge portion 26 to the downwardly depending ring 25 of the resealing overcap 20 is between 30° and 45°. The resealing overcap may be constructed of any suitable material having the characteristics described above and preferably is constructed of plastic and is injection molded so as to provide the downwardly depending flexible ring 25 which allows flexure and snap fitting around the bead 18 of the end ring 17 of the top end closure 15 of the container 10. [0017] Thus, the present invention has provided an improved resealing overcap for use on a container having an easy-opened top end closure and which is constructed to provide for resealing of the container after the top end closure has been opened to gain access to the product within the container.

[0018] In the drawings and specification, there has been set forth a preferred embodiment of this invention and, although specific terms are employed, these terms are used in a generic and descriptive sense only and not for purposes of limitation. The scope of the invention is set forth in the following claims.

Claims

- 1. In a container, adapted to be filled with powdered products, dry food products and the like, comprising a generally cylindrical container body defining top and bottom opposite end portions, top and bottom end closures attached respectively to said top and bottom container body end portions for closing and sealing the container with product therein, wherein said top end closure is of the easy-open type and comprises an end ring having an outside circular peripheral area seamed to said container body top end portion and having an inside circular peripheral area in the form of a downwardly and inwardly curved bead for defining a central opening of desired size to allow access therethrough to the interior of said container, and wherein said top end closure further comprises an easy-open membrane patch of sufficient size to cover said central opening and which is attached to said end ring for allowing detachment and removal when it is desired to open said container to obtain access to the product therein; the improvement of:
 - a resealing overcap constructed for fitting over said container body top end portion and said top end closure before and after opening of said container and for snap fitting into said central opening of said top end closure after opening of said container and removal of said easy-open membrane patch for resealing of said container; said overcap comprising a circular central portion having an outer

periphery and extending radially of said container for covering said container top end portion, a downwardly depending flange portion integrally extending from and around said central portion outer peripheral area and axially of said container body and having an inside diameter approximately equal to an outside diameter of said container body to provide a friction fit around said end ring seamed to said container body top end portion, and a downwardly depending flexible ring portion integrally extending from an inside surface of said overcap central portion axially of said container body to define a leading edge portion positioned over said inside peripheral area of said top end closure end ring before opening of said container and adapted to flex and snap fit around said downwardly and inwardly curved bead forming said inside peripheral area of said top end closure upon opening of said container and removal of said easy-open membrane patch.

- 2. In a container, as set forth in claim 1, in which said leading edge portion of said downwardly depending flexible ring of said resealing overcap is of a diameter larger than a diameter of said inside peripheral area of said container top end closure end ring and extends outwardly of said overcap at a predetermined angle to said overcap downwardly depending flexible ring.
- In a container, as set forth in claim 2, in which said predetermined angle of said leading edge portion to said downwardly depending flexible ring of said overcap is between 30° and 45°.
- 4. In a container, as set forth in claim 1, 2 or 3, in which said overcap comprises an injection molded plastic overcap.
 - 5. A resealing overcap adapted to fit over a top end of a cylindrical container and an easy-open top end closure thereon which includes an end ring having an inside circular downwardly and inwardly curved bead defining a central opening which is closed by a removable membrane positioned thereon; said overcap comprising:
 - a circular central portion having an outer periphery and adapted to extend radially of the container when positioned thereon for covering the container top end closure;
 - a downwardly depending flange portion integrally extending from and around said central portion outer peripheral area and adapted to extend axially of the cylindrical container when positioned thereon and having an inside diameter approximately equal to an outside diameter of the cylindrical container to provide a friction fit around the container top end closure

when positioned thereon; and a downwardly depending flexible ring portion integrally extending from an inside surface of said overcap central portion and adapted to extend axially of the cylindrical container when positioned thereon to define a leading edge portion adapted to be positioned over the inside circular bead of the container end closure when positioned thereon before opening of the container and adapted to flex and snap fit around the downwardly and inwardly curved bead of the end ring of the container end closure upon opening of the container and removal of the easy-open membrane patch for resealing of the container.

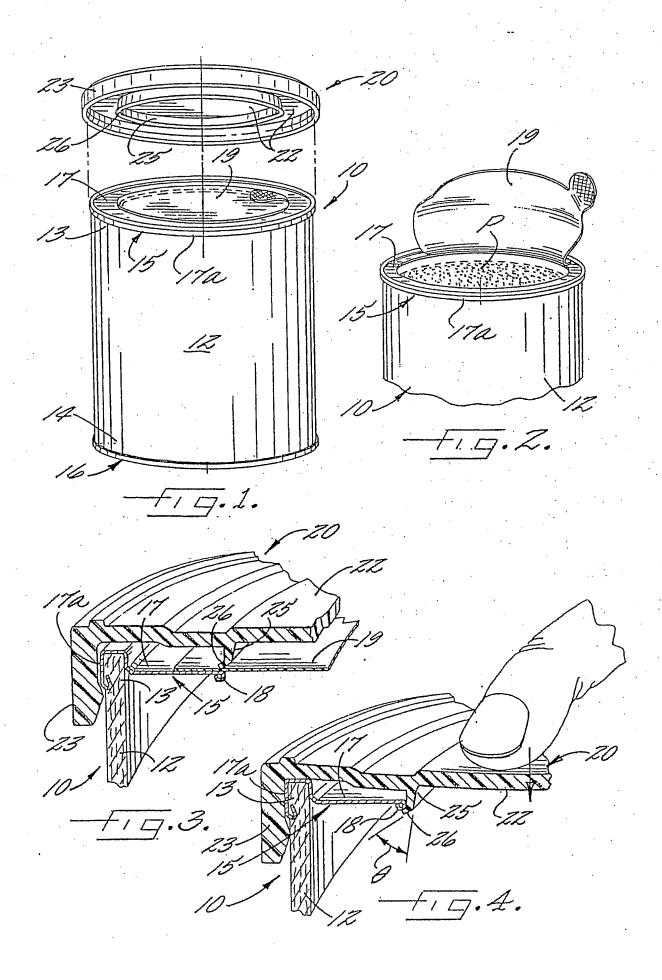
6. A resealing overcap, as set forth in claim 5, in which said leading edge portion of said flexible ring of said overcap is of a diameter larger than the central opening of the container top end closure and extends outwardly of said overcap at a predetermined angle of between 30° and 45° to said overcap downwardly depending flexible ring.

7. A resealing overcap, as set forth in claim 5 or 6, in 25 which said overcap comprises an injection molded plastic overcap.

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EUROPEAN SEARCH REPORT

Application Number

EP 00 31 0164

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Category	Citation of document with Ir of relevant pass	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int.CI.7)	
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Υ	21 October 1969 (19 * page 2, line 30 - figures 1,2 *	page 4, line 19;	1-4,6,7	
Υ	EP 0 357 276 A (GEN 7 March 1990 (1990- * column 5, line 23	1-4,6,7		
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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ :		(11) International Publication Number:	WO 00/26878
G08B 13/14	A1	(43) International Publication Date:	11 May 2000 (11.05.00)

(21) International Application Number:

PCT/US99/24104

(22) International Filing Date:

14 October 1999 (14.10.99)

(30) Priority Data:

09/182,022

29 October 1998 (29.10.98) US

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(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

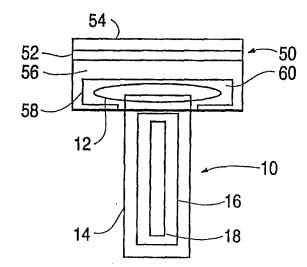
Published

With international search report.

(54) Title: CAP WITH INTEGRATED EAS MARKER

(57) Abstract

A cap (50) for a bottle to be protected from theft includes a covering portion (52) for covering an opening of the bottle and an electronic article surveillance ("EAS") marker (10). The covering portion has a surface (54) and a wall (56) that extends downward from the surface and along a circumference of the surface. The EAS marker comprises a circular disk (12) positioned within a cavity (60) defined by an inner surface (58) of the wall of the covering portion and a marker element attached to and extending downward from the circular disk. The marker element is insertable through the opening of the bottle and provides a signal that is detectable by an electronic article surveillance system.



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CAP WITH INTEGRATED EAS MARKER

FIELD OF THE INVENTION

This invention relates generally to electronic article surveillance ("EAS"), and more particularly, to techniques for securing EAS markers to goods that are to be protected from theft.

BACKGROUND OF THE INVENTION

It is known to provide electronic article surveillance systems in which detecting devices are installed at the exits of retail stores. EAS markers that are detectable by the detecting devices are secured to articles of merchandise that are to be protected from theft. When a customer pays for an article of merchandise, the EAS marker secured to the article of merchandise is either removed or deactivated according to known techniques. The article can then be carried past the detecting devices without actuating an alarm.

Articles of merchandise in the form of bottles, and particularly wine bottles or other beverage bottles, present particular challenges in terms of securing EAS markers to the bottles. For the most part, EAS markers are provided in a form that is suitable for attachment to a large flat surface, such as the cover of a jewel case for a compact disc, or in a device that is attachable to soft goods, such as clothing. A wine bottle, however, lacks flat surfaces to which conventional EAS markers can be readily attached and does not provide for easy attachment as with clothing.

French Patent Application No. 2,703,659 (" '659 French Application") proposes a bottle cap arrangement for a wine bottle, with an anti-theft circuit mounted in the cap arrangement at a position above the opening of the bottle. More particularly, the anti-theft circuit is sandwiched between the upper surface of a disk that covers the opening of the bottle and a circular base of a capsule that covers the disk as well as the neck of the bottle. This anti-theft circuit shown in the '659 French Application, however, appears to be incompatible with the most widely installed types of EAS detection equipment, and also appears to be of a type that would be masked and rendered ineffective if the top of the bottle is covered with metal foil or metal cap, as is often done with champagne and wine. Furthermore, the anti-theft circuit is limited to its placement between the disk covering the opening of the bottle and the circular base of the capsule.

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French Patent No. 95 12402 ("'402 Patent"), issued to Dameme and commonly assigned with the present application, also proposes an arrangement for protecting a bottle from theft, but unlike the '659 French Application, uses an electronic article surveillance marker mounted in a bottle cover covering the top and neck of a bottle. Such a bottle protection arrangement, however, may not protect a bottle from theft if the bottle cover is torn revealing the EAS marker or the bottle cover and marker are removed from the bottle. This arrangement also is not suitable for protecting bottles that are sold without covers.

It is accordingly an object of the invention to provide an improved technique for securing an EAS marker to a bottle to be protected from theft.

It is a more particular object of the invention to provide a technique for integrating in a cap or lid of a bottle an EAS marker that is compatible with an installed base of EAS detection equipment.

It is a further object of the invention to provide a technique for integrating an EAS marker in a cap or lid of a bottle so as to decrease the marker's visibility.

SUMMARY OF THE INVENTION

In accordance with the principles of the present invention, the above and other objectives are realized in a cap for a bottle to be protected from theft, the cap comprising a covering portion and an electronic article surveillance ("EAS") marker. The covering portion has a surface and a wall that extends downward from the surface and along a segment of the surface. The EAS marker comprises an attachment portion or a disk-shaped member for attaching the EAS marker to the covering portion and a marker element attached to the disk-shaped member for providing a signal that is detectable by an electronic article surveillance system. The disk-shaped member is positioned within a cavity defined by the inner surface of the wall of the covering portion and the marker element is positioned perpendicular to and extends downward from the disk-shaped member. The bottle itself has an opening at the top and a neck portion that extends downward from the opening. The marker element is insertable through the opening into the neck portion of the bottle.

The marker element of the EAS marker provided according to the above aspects of the invention may be a magnetostrictive member and a biasing member mounted spaced apart from the magnetostrictive material. The biasing member, when magnetically biased, causes the magnetostrictive member to be mechanically resonant when exposed to an alternating electromagnetic field generated at a selected frequency by an electronic article surveillance

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system. The marker element of the present invention may also include a continuous strip of magnetic material or a marker suitable for radio frequency or microwave EAS systems.

In a further aspect of the invention, the disk-shaped member of the EAS marker is attached to a bottom surface of the covering portion and the marker element attached to the disk-shaped member is insertable through the opening into the neck portion of the bottle.

In the present invention, there is also provided a method of forming a cap to cover an opening of a bottle to be protected from theft. The method comprises the steps of providing a covering portion for covering the opening of the bottle and positioning an attachment portion of an EAS marker within a cavity defined by an inner surface of a wall of the covering portion. The EAS marker further comprises a marker element that is attached to and extends downward from the attachment portion for insertion through the opening of the bottle in order to provide a signal that is detectable by an electronic article surveillance system.

Another method of the present invention provides for an attachment portion of the EAS marker to be attached to a bottom surface of a covering portion of the cap with the marker element attached to the disk-shaped member being insertable through the opening into the neck portion of the bottle.

BRIEF DESCRIPTION OF THE DRAWINGS

The above and other features and aspects of the present invention will become more apparent upon reading the following detailed description in conjunction with the accompanying drawings, in which:

- FIG. 1 shows a schematic side view of the EAS marker of the present invention;
- FIG. 2 shows a schematic side view of the EAS marker of FIG. 1 incorporated in a cap of a bottle;
- FIG. 3 shows a schematic side view of a bottle with the EAS marker and cap of FIG. 2 inserted in the neck of the bottle;
- FIG. 4 shows a schematic side view of the EAS marker of FIG. 1 incorporated in another cap of a bottle;
- FIG. 5 shows a schematic side view of another bottle with the EAS marker and cap of FIG. 4 inserted in the neck of another bottle; and
- FIG. 6 shows a schematic view of an installation of conventional EAS detection equipment.

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DETAILED DESCRIPTION

FIGS. 1-5 show the EAS marker 10 in accordance with the principles of the present invention. In the present illustrative case, the EAS marker 10 is attachable to a variety of different types of caps or lids for bottles or containers. Indeed, the EAS marker 10 is easily integrated into or attached to a cap and then inserted through the opening of the bottle into the neck portion of a bottle to permit concealment of the EAS marker 10 to shoppers in an EAS environment.

This ease of attachment and concealment of the EAS marker 10 is based upon its T-shaped structure (as seen in a side view). With reference to FIG. 1, the EAS marker 10 comprises an attachment portion or disk-shaped member 12 and a box-like housing or support 14 in which is disposed an active marker element comprising an active or magneto-mechanical element 16 and magnetic biasing element 18. These elements in the housing 14 cooperate to generate a detectable signal in the manner disclosed in U.S. Patent No. 4,510,489 issued to Anderson et al., the teachings of which are incorporated herein by reference. As illustrated in FIG. 1, the disk-shaped member 12 has top and bottom surfaces 20 and 22 with an edge 24 of the housing 14 being attached to the disk-shaped member's bottom surface 22. The housing 14 with the elements 16 and 18 therein is positioned perpendicular to the disk-shaped member's bottom surface 22 and extends downward to form the T-shaped structure of the EAS marker 10 as shown in FIG. 1.

In particular, the positioning of the disk-shaped member 12 relative to the housing 14 allows for integration of the marker 10 with the cap 50 as shown in FIG. 2. As FIGS. 2 and 3 illustrate, the cap has a covering portion 52 that covers an opening 64 of the bottle 62. The covering portion 52 has a surface 54 and a wall 56 extending downward from the surface 54 and along a circumference of the surface 54. The wall 56 has an inner surface 58. The EAS marker 10 is integrated in the cap 50 by the disk-shaped member 12 being inserted into a cavity 60 defined by the inner surface 58 of the wall 56 of the covering portion 52. The disk-shaped member 12 having a slightly smaller diameter than compared to the diameter of the cap 50 allows for such insertion and integration. With the disk-shaped member 12 of the EAS marker 10 inserted in the cavity 60, as shown in FIG. 2, the housing 14 with the magneto-mechanical element 16 and magnetic biasing element 18 disposed therein extends perpendicular to and downward from the disk-shaped member 12.

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FIG. 3 shows the EAS marker 10 integrated in the cap 50 and being inserted through an opening 64 into a neck portion 66 of the bottle 62. Accordingly, when the cap 50 is positioned on the bottle 62, the EAS marker 10 extends vertically downwardly in the neck portion 66 of the bottle 62.

FIGS. 4 and 5 illustrate another type of cap and bottle for use with the EAS marker 10. FIG. 4 illustrates the EAS marker 10 attached to a cap 72 having a covering portion 74 with top and bottom surfaces 76 and 78. In this illustrated embodiment, the top surface 20 of the disk-shaped member 12 of the EAS marker 10 is attached or secured adjacent to the bottom surface 78 of the covering portion 74. The disk-shaped member 12 provides means for suspending the housing 14 of the marker 10 from the covering portion's bottom surface 78. The housing 14 extends substantially vertically downward from the disk-shaped member 12. The marker element disposed in the housing 14, is then insertable through an opening 80 into a neck portion 82 of the bottle 70 as shown in FIG. 5.

In order to show an example of detection equipment for use with the present invention, FIG. 6 is provided as an illustration with detection equipment 100 including antenna pedestals 102 and 104 defining therebetween a passage 106 for detecting unauthorized removal of a bottle with the EAS marker 10 integrated therewith.

The EAS marker 10 of the present invention, however, is not limited to its present configuration but can be a variety of different shapes and sizes. For example, an attachment portion of the EAS marker 10 can be square, rectangular or otherwise polygonal, etc., instead of disk-shaped (circular). In addition to or instead of pressure fitting the attachment portion to the bottle cap, adhesives, tape, epoxy or connectors for securing the EAS marker 10 to the cap may be used. The housing 14 may also be a variety of shapes and sizes that accommodate or support a detectable marker element and that is insertable into the opening or neck of a bottle or container.

In addition, although the present invention has been described herein with reference to a magneto-mechanical type of marker disclosed in the aforesaid Anderson et al. patent, it is contemplated according to the present invention to integrate marker elements of other types and/or other shapes in the housing 14 of the EAS marker 10. For example, it is contemplated to place in the housing 14 an element or ribbon of highly permeable magnetic material (like that disclosed in U.S. Patent No. 4,686,516 issued to Humphrey and commonly assigned with the present application) or an integrated circuit marker capable of generating a multi-bit

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identification signal. The latter type of marker would be especially useful in applications in which it was desired to prevent counterfeiting as well as to deter theft. Accordingly, the multi-bit marker may advantageously be used in connection with expensive brands of champagne or perfume. It is also contemplated to apply the present invention to markers comprising a resonant circuit that is suitable for radio frequency or microwave EAS systems.

Although the invention has, up to this point, primarily been described in an embodiment suitable for use with a wine or champagne bottle, the invention can also be applied to caps for bottles containing other types of beverages, and on bottles containing fluids that are not beverages, including condiments or perfume, for example. Moreover, the invention may be used with bottles that contain non-fluid substances, including foods, pills or powders. The term "bottle", as used herein, should be understood to include containers made of plastic or other materials, in addition to glass, and is not limited to containers having a circular profile in a horizontal cross-section. Containers having, for example, horizontal cross-sections that are rectangular in profile are also to be embraced within the term "bottle", as well as any number of other geometric cross-sections and dimensions.

In all cases it is understood that the above-described arrangements and structures are merely illustrative of the many possible specific embodiments which represent applications of the present invention. Numerous and varied other configurations, can be readily devised in accordance with the principles of the present invention without departing from the spirit and scope of the invention.

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What is claimed is:

1. A cap for a bottle to be protected from theft, said cap comprising:

a covering portion for covering an opening of the bottle, the covering portion having a surface and a wall extending downward from the surface and along a circumference of said surface, said wall having an inner surface; and

an electronic article surveillance ("EAS") marker comprising an attachment portion and a marker element, said attachment portion positioned within a cavity defined by said inner surface of said wall and said marker element attached to and extending downward from the attachment portion, said marker element for providing a signal that is detectable by an electronic article surveillance system.

- 2. A cap according to claim 1, wherein said attachment portion is a disk-shaped member that is positioned in the cavity of the covering portion.
- 3. A cap according to claim 2, wherein said marker element comprises a magnetostrictive member and a biasing member mounted spaced apart from said magnetostrictive member, said biasing member, when magnetically biased, for causing said magnetostrictive member to be mechanically resonant when exposed to an alternating electromagnetic field generated at a selected frequency by an electronic article surveillance system.
- 4. A cap according to claim 3, wherein said EAS marker further comprises a housing for said magnetostrictive member and said biasing member.
- 5. A cap according to claim 2, wherein said marker element comprises a magnetic element.
- 6. A cap according to claim 5, wherein said EAS marker further comprises a support for said magnetic element.
- 7. A cap according to claim 2, wherein said marker element comprises a resonant circuit adapted to be responsive to radio frequency or microwave frequency signals.
- 8. A cap according to claim 7, wherein said EAS marker further comprises a support for said resonant circuit.
 - 9. A cap according to claim 1, wherein said EAS marker is T-shaped.
- 10. A cap for a bottle to be protected from theft, said cap comprising:

 a covering portion for covering an opening of the bottle, the covering portion having top and bottom surfaces; and

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an electronic article surveillance ("EAS") marker comprising an attachment portion and a marker element, said attachment portion attached to said bottom surface of the covering portion and said marker element attached to and extending downward from the attachment portion, said marker element for providing a signal that is detectable by an electronic article surveillance system and being insertable through the opening of the bottle.

- 11. A cap according to claim 10, wherein said attachment portion is a disk-shaped member having top and bottom disk surfaces, said top disk surface being attached to the bottom surface of the covering portion.
- 12. A cap according to claim 11, wherein said marker element comprises a magnetostrictive member and a biasing member mounted spaced apart from said magnetostrictive member, said biasing member, when magnetically biased, for causing said magnetostrictive member to be mechanically resonant when exposed to an alternating electromagnetic field generated at a selected frequency by an electronic article surveillance system.
- 13. A cap according to claim 12, wherein said EAS marker further comprises a housing for said magnetostrictive member and said biasing member.
- 14. A cap according to claim 11, wherein said marker element comprises a magnetic element.
- 15. A cap according to claim 14, wherein said EAS marker further comprises a support for said magnetic element.
- 16. A cap according to claim 11, wherein said marker element comprises a resonant circuit adapted to be responsive to radio frequency or microwave frequency signals.
- 17. A cap according to claim 16, wherein said EAS marker further comprises a support for said resonant circuit.
 - 18. A cap according to claim 10, wherein said EAS marker is T-shaped.
- 19. An electronic article surveillance marker comprising:

 an active element for providing a signal that is detectable by an electronic surveillance system;
 - a housing in which said active element is contained; and means for suspending said housing from a bottom surface of a bottle cap.
- 20. A marker according to claim 19, wherein said housing extends substantially vertically downward from said means for suspending.

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21. A marker according to claim 19, wherein said means for suspending includes a disk-shaped member for being secured adjacent to said bottom surface of said bottle cap.

- 22. A marker according to claim 19, wherein said active element is a magnetostrictive member, said marker further comprising a biasing member mounted to said housing, said biasing member, when magnetically biased, for causing said magnetostrictive member to be mechanically resonant when exposed to an alternating electromagnetic field generated at a selected frequency by an electronic article surveillance system.
- 23. A method of forming a cap to cover an opening of a bottle to be protected from theft, the method comprising the steps of:
- (a) providing a covering portion for covering the opening of the bottle, said covering portion having a surface and a wall extending downward from the surface and along a circumference of said surface, said wall having an inner surface; and
- (b) positioning an attachment portion of an electronic article surveillance ("EAS") marker within a cavity defined by said inner surface of said wall of the covering portion, said EAS marker further comprising a marker element attached to and extending downward from the attachment portion for insertion through the opening of the bottle.
- 24. A method according to claim 23, wherein said marker element comprises a magnetostrictive member and a biasing member mounted spaced apart from said magnetostrictive member, said biasing member, when magnetically biased, for causing said magnetostrictive member to be mechanically resonant when exposed to an alternating electromagnetic field generated at a selected frequency by an electronic article surveillance system.
- 25. A method according to claim 23, wherein said marker element comprises a magnetic element.
- 26. A method according to claim 23, wherein said marker element comprises a resonant circuit adapted to be responsive to radio frequency or microwave frequency signals.
- 27. A method of forming a cap to cover an opening of a bottle to be protected from theft, the method comprising the steps of:
- (a) providing a covering portion for covering the opening of the bottle, said covering portion having top and bottom surfaces; and
- (b) attaching an attachment portion of an electronic article surveillance ("EAS") marker to the bottom surface of the covering portion, said EAS marker further

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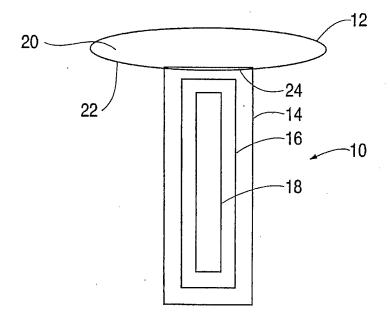
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comprising a marker element attached to and extending downward from the attachment portion for insertion through the opening of the bottle.

- 28. A method according to claim 27, wherein said marker element comprises a magnetostrictive member and a biasing member mounted spaced apart from said magnetostrictive member, said biasing member, when magnetically biased, for causing said magnetostrictive member to be mechanically resonant when exposed to an alternating electromagnetic field generated at a selected frequency by an electronic article surveillance system.
- 29. A method according to claim 27, wherein said marker element comprises a magnetic element.
- 30. A method according to claim 27, wherein said marker element comprises a resonant circuit adapted to be responsive to radio frequency or microwave frequency signals.
- 31. A method of securing an EAS marker to a bottle to be protected from theft, said bottle having an opening at a top of the bottle and a neck portion that extends downwardly from said opening, the method comprising the steps of:
 - (a) securing said EAS marker to a bottom surface of a bottle cap;
 - (b) inserting said EAS marker into the neck portion of said bottle; and
- (c) installing said bottle cap on said bottle in a position to cover said opening of said bottle.
- 32. A method according to claim 31, wherein said EAS marker further comprises a marker element.
- 33. A method according to claim 32, wherein said marker element comprises a magnetostrictive member and a biasing member mounted spaced apart from said magnetostrictive member, said biasing member, when magnetically biased, for causing said magnetostrictive member to be mechanically resonant when exposed to an alternating electromagnetic field generated at a selected frequency by an electronic article surveillance system.
- 34. A method according to claim 32, wherein said marker element comprises a magnetic element.
- 35. A method according to claim 32, wherein said marker element comprises a resonant circuit adapted to be responsive to radio frequency or microwave frequency signals.

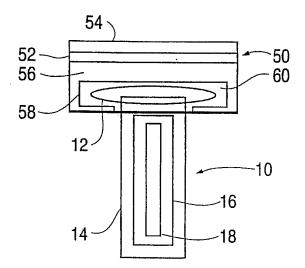
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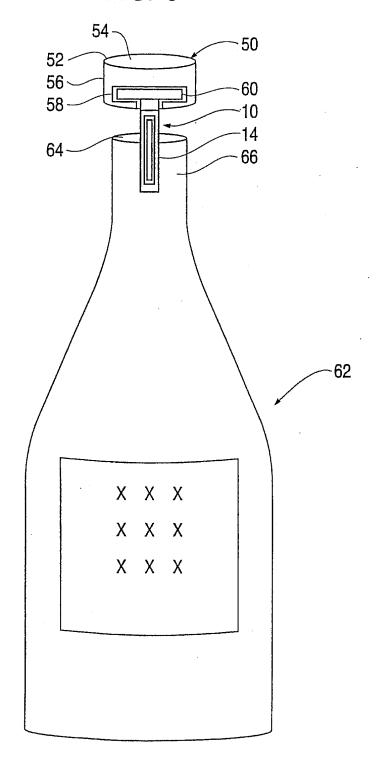
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FIG. 2

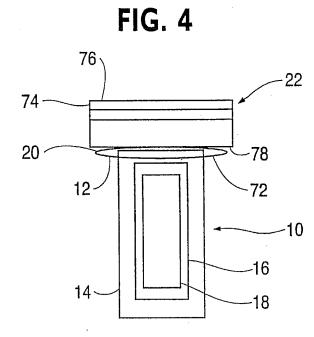


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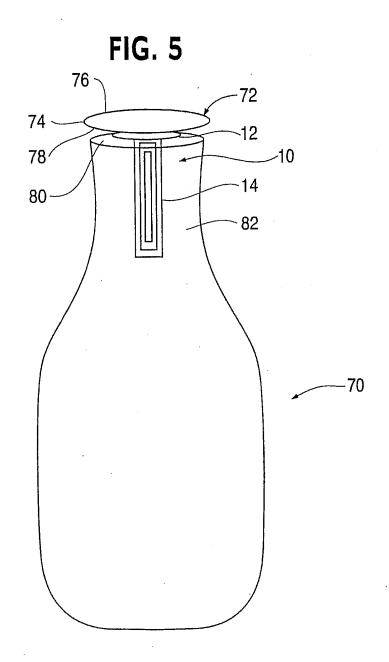
FIG. 3



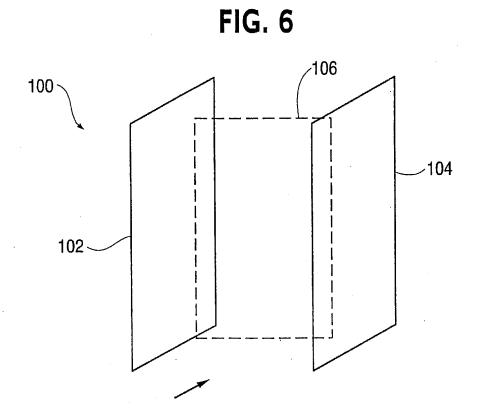
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INTERNATIONAL SEARCH REPORT

International application No. PCT/US99/24104

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A. CLASSIFICATION OF SUBJECT MATTER IPC(6) :G08B 13/14							
US CL :340/572.8 According to International Patent Classification (IPC) or to both national classification and IPC							
B. FIEL	DS SEARCHED						
Minimum do	cumentation searched (classification system followed	by classification symbols)					
U.S. : 3	40/572.8, 572.5, 572.6, 572.7, 572.9, 551						
Documentati	Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched						
Electronic d WEST/BR	ata base consulted during the international search (nam S	e of data base and, where practicable,	search terms used)				
C. DOC	UMENTS CONSIDERED TO BE RELEVANT						
Category*	Citation of document, with indication, where appr	ropriate, of the relevant passages	Relevant to claim No.				
Y	US 5,602,530 A (HOLMGREN) 11 Feb 67.	oruary 1997, col. 3, lns. 63-	1-35				
Y	Y US 5,625,347 A (MACLEAN et al) 29 April 1997, figure 2.						
Y	US 5,777,553 A (PERREAU et al) 07 60.	3, 4, 7, 8, 12, 13, 16, 17, 22, 24, 26, 28, 30, 33-35					
A	US 4,813,564 A (COOPER et al) 21 M	1-35					
A	US 5,729,200 A (COPELAND et al) 1	1-35					
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Furt	Further documents are listed in the continuation of Box C. See patent family annex.						
• S	ternational filing date or priority dication but cited to understand e invention						
E e	to be of particular relevance "X" goodment of particular relevance; the state of t						
0	he claimed invention cannot be a step when the document is						
n	"O" document referring to an oral disclosure, use, exhibition or other combined with one or more other a being obvious to a person skilled it						
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Date of the actual completion of the international search 07 JANUARY 2000 Date of mailing of the international search report 16 FEB 2000							
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230 Authorized officer JOHN TWEEL, JR. Telephone No. (703) 308-7826							

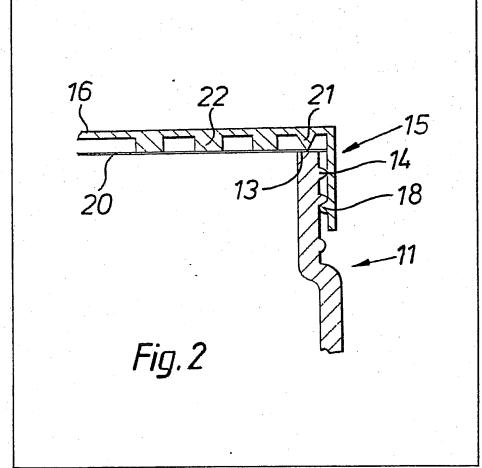
(12) UK Patent Application (19) GB (11)

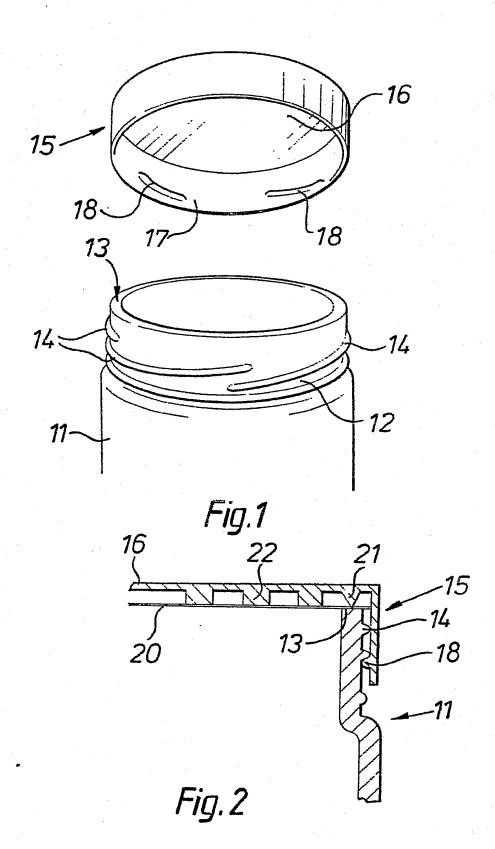
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- (21) Application No 8317923
- (22) Date of filing 1 Jul 1983
- (30) Priority data
- (31) 8220130
- (32) 10 Jul 1982
- (33) United Kingdom (GB)
- (43) Application published 1 Feb 1984
- (51) INT CL3
- B65D 51/20
- (52) Domestic classification B8T 120B 13A CA CC U1S 1105 B8T
- (56) Documents cited GB 1231208 GB 1153063 GB 1087437
- (58) Field of search B8T
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(54) Container sealing devices and method of sealing containers

(57) A cap (15) has screw-thread portions (18) for screw engagement with threads (14) on a jar (11). The inner surface of the top of the cap is formed with ribs (22) and an annular sealing member (21). A frangible membrane (20) is weakly adhered to the ribs and the sealing member and is more strongly adhered to the rim (13) of the jar. The cap can be unscrewed from the jar leaving the membrane in situ sealing the jar. After removal of the membrane, the sealing member seals with the jar rim.





SPECIFICATION

Container sealing devices and method of sealing containers

5 This invention is concerned with sealing of containers, such as jars.

Jars are commonly used for storing foodstuffs and the jar is usually closed by a cap screwed onto the jar. A cap liner is usually provided to seal with the jar rim.

10 For some foodstuffs, such as "instant" coffee, which is deliquescent, it is usual to seal the jar mouth with a frangible membrane, which is adhered to the rim. A liner is also provided and is held by formations in the cap. The membrane is ripped away for access to the contents, after which sealing is effected by the liner.

A sheet of membrane material is usually adhered with a weak bond to a sheet of the liner material and discs are cut from the combined sheet to the size of the appropriate cap and jar. A disc is engaged with the

20 formations of the cap and glue is applied to the jar rim.
 The cap is then screwed down, so that the membrane engages the glue. A stronger bond is provided

between the membrane and the jar rim than that between the membrane and the liner and this latter

25 bond is weaker than the grip of the liner by the cap formations. As a result, on unscrewing the cap, the liner comes away from the membrane.

The provision of a liner is expensive and it would be advantageous to dispense with the liner. This, howev30 er, creates the problems of how to provide for a seal between the cap and the jar rim and how to hold the membrane (which is flexible) in the cap to permit engagement of the membrane with the jar rim for adhesion thereto, when the cap is screwed down on the jar.

In accordance with this invention, there is provided a cap having a top and a skirt adapted internally to allow repetative engagement with and release from a container and a flexible sealing membrane for adherance to the rim of the container, wherein the membrane is weakly adhered directly to the internal face of the top of the cap. The cap preferably has a formation for sealing with the container rim when the membrane has been removed.

Adherence of the membrane to the cap itself presents a problem, since such caps are usually made of polypropylene to which it is difficult to provide reliably a weak bond.

Preferably, the material serving to adhere the membrane to the cap is a waxy material which is heat-sealed to the cap.

The invention also resides in a cap and container assembly, wherein the container has a rim and the cap has a top and a skirt, the skirt being engaged with the container for release therefrom and repetitive engagement therewith, and a sealing membrane adhered to the internal face of the top of the cap and also adhered to the container rim, the bond with the cap being weaker than the bond with the rim, whereby the cap can be removed from the container leaving the membrane adhered to the rim.

The invention also resides in a method of mounting a membrane in a cap, which cap has a top and a skirt adapted for repetitive engagement with and release from a container, said mounting being such that on engaging the cap on a container, the membrane may adhere to the container rim by means of adhesive, such that on removal of the cap, the membrane is released from the cap and remains adhered to the rim, the method comprising applying waxy material to one face of the membrane and heat-sealing the membrane directly to the interior surface of the top of the cap by

Reference is now made to the accompanying 75 drawings, wherein:

means of the waxy material.

Figure 1 shows a part of a jar and a cap adapted for attachment thereto; and

Figure 2 is a detailed view of part of the cap and the jar rim, with the cap attached to the jar.

Figure 1 shows a glass jar 11 having a neck 12 provided with a rim 13 and having external screw threads 14. A cap 15, moulded from polypropylene, has a top 16 and a skirt 17. The skirt has internal screw threads 18 for engaging the threads 14 of the jar.

A membrane 20 (Figure 2) is adhered in the cap directly to the internal surface of the top 16. The membrane is of disc form and is almost of the same diameter as the internal diameter of the top 16.

When the cap is first attached to the jar, after the jar 90 has been filled, the rim 13 of the jar is first coated with adhesive. The cap is then screwed down onto the jar, so that the membrane adheres to the rim.

The bond between the top 16 of the cap and the membrane is relatively weak, so that, on unscrewing 95 of the cap, the bond is broken, leaving the membrane adhered to the rim. The membrane is frangible and can be ripped away from the jar mouth for access to the contents. The membrane may be composed of a metal foil or a waxed paper, for example.

100 The cap can be re-attached to the jar and has an annular sealing member 21, on the internal surface of the top 16 of the cap, which member resiliently engages the rim 13.

Whithin the annular sealing member, the top of the cap has ribs 22, which may be linear or annular, and which are positioned so as not to engage with the jar rim. The membrane is initially adhered to the sealing member 21 and the ribs 22.

The adherence is provided by a waxy material a
110 coating of which is initially applied to one surface of
the membrane. This coating is heated prior to being
applied to the top of the cap, so that, on cooling, the
coating adheres to the top of the cap.
CLAIMS

- 11. A cap having a top and a skirt adapted internally to allow repetitive engagement with and release from a container and a flexible sealing membrane for adherence to the rim of the container, wherein the membrane is weakly adhered directly to the internal 120 face of the top of the cap.
 - 2. A cap according to Claim 1 including a waxy material heat-sealed to the cap and serving to adhere the membrane to the cap.

3. A cap according to Claim 2, wherein the cap is a polypropylene moulding.

4. A cap according to any preceding claim, wherein the internal face of the top of the cap has ribs to5 which the membrane is weakly adhered.

5. A cap according to any preceding claim, including a formation for sealing with the container rim when the membrane has been removed.

A cap constructed substantially as herein de scribed with referece to the accompanying drawings.

- 7. A cap and container assembly, wherein the container has a rim and the cap has a top and a skirt, the skirt being engaged with the container for release therefrom and repetitive engagement therewith, and a sealing membrane adhered to the internal face of the top of the cap and also adhered to the container rim, the bond with the cap being weaker than the bond with the rim, whereby the cap can be removed from the container leaving the membrane adhered to the rim.
- 8. A cap and container assembly according to Claim 6, wherein the cap is constructed according to Claim 2, 3, 4, 5 or 6.
- A method of mounting a membrane in a cap, which cap has a top and a skirt adapted for repetitive engagement with and release from a container, the mounting being such that on engaging the cap on a container, the membrane may adhere to the container rim by means of adhesive, such that on removal of the cap, the membrane is released from the cap and remains adhered to the rim, the method comprising applying waxy material to one face of the membrane and heat-sealing the membrane directly to the interior surface of the top of the cap by means of the waxy
- 35 10. A method of mounting a membrane in a cap, substantially as herein described with reference to the accompanying drawings.

material.

Printed for Her Majesty's Stationery Office by The Tweeddale Press Ltd., Berwick-upon-Tweed, 1984. Published at the Patent Office, 25 Southampton Buildings, London WC2A 1AY, from which copies may be obtained.



(11) EP 1 266 839 A2

(12)

EUROPEAN PATENT APPLICATION

(43) Date of publication:18.12,2002 Bulletin 2002/51

(51) Int CI.7: **B65D 47/10**

(21) Application number: 02254152.8

(22) Date of filing: 13.06.2002

(84) Designated Contracting States:
AT BE CH CY DE DK ES FI FR GB GR IE IT LI LU
MC NL PT SE TR
Designated Extension States:
AL LT LV MK RO SI

(30) Priority: 13.06.2001 GB 0114410

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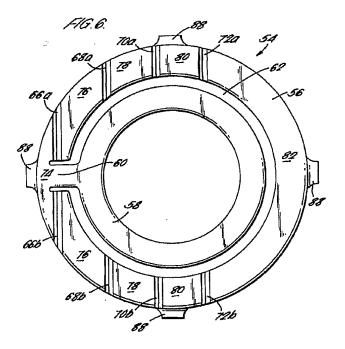
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(54) Pull-tab

(57) There is described a pull-tab for use in removing a membrane adhered to a container (32). The container (32) defines an opening (40) having a periphery and the membrane overlies the opening (40) and is adhered to the periphery so as to prevent access to the contents of the container (32) via the opening (40) prior to the removal of the membrane. The pull-tab (54) comprises a peripheral portion having a shape correspond-

ing to at least a part of the periphery of the opening (40) and is joined to the membrane in a region approximate that part. A tab portion (58) is joined to the peripheral portion (56). The peripheral portion (56) comprises means (66,68,70,72) to restrict a pulling force applied to the tab portion (58) from being substantially transmitted to all but a sector of the peripheral portion until such time as the membrane approximate that sector has been pulled away from the container (32).



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Description

[0001] The present invention relates to a pull-tab for use in releasing a membrane adhered to a container, the container defining an opening having a periphery and the membrane overlying the opening and being adhered to the periphery so as to prevent access to the contents of the container via the opening prior to the release of the membrane. The present invention also relates to a pull-tab in combination with such a membrane and to a pull-tab and membrane in combination with such a container.

[0002] It is known to package goods in plastics containers in which the plastics container is provided with an opening which is overlain by a plastics cap or other closure to provide resealable access to the contents of the container via the opening. It is also known to provide such a cap or closure with a foil liner which covers the opening and which is at least initially adhered to the container. The provision of such a liner serves not only to retain the freshness of the product packaged within the container but also serves to provide the consumer with evidence as to whether the container has been subject to unauthorised tampering since, under such circumstances, the foil liner will either no longer be adhered to the container or else will be compromised in some other way.

[0003] Typically, as shown in Figure 1, the foil liner 10 comprises a metallic substrate 12 which is coated on each of its opposing surfaces with a respective layer of adhesive 14,16. Each of the layers of adhesive 14,16 in turn adhere the metallic substrate 12 to a respective layer of polyethylene material 18,20. One of the layers of polyethylene material 18 is also bonded to a form layer 22 which provides a non-stick coating to one side of the foil liner 10.

[0004] The foil liner is adhered to the container by placing it over the opening with the form layer uppermost and holding it in close contact with the rim. The foil liner is then exposed to a time varying magnetic field which gives rise to eddy currents within the electrically conductive metallic substrate 12 and the generation of heat. This heat in turn melts the respective polyethylene layers 18 and 20 and bonds the foil liner to the container. [0005] In order to first gain access to the contents of the container the foil liner must be removed and discarded. To this end it is known to provide the liner with a tab 24 which is not adhered to the container and which projects outwardly away from the underlying opening as shown in Figure 1. In order to remove the foil liner it is intended that the consumer pull the tab in such a way as to overcome the bond between the foil liner and the container. However, such tabs are small and typically difficult to grasp, particularly for the elderly and those with limited dexterity, and as a result it is has not always

[0006] In an attempt to overcome the difficulties as-

reliable manner.

been possible to remove the foil liner in an easy and

sociated with the tab arrangement shown in Figure 1, foil liners have also been designed of the type shown in Figure 2 which present a semi-circular pull-tab of increased surface area and which are consequently easier to grasp. The foil liners are stamped from a web of material shown in Figure 3 which, as before, comprises a metallic substrate 12 sandwiched between two layers of adhesive 14 and 16 and, outside that, two layers of polyethylene material 18 and 20. One of the layers of polyethylene material 18 is again provided with a form layer 22 and it is here that the foil liner of Figure 2 differs from that of Figure 1. The form layer 22 comprises a laminate of two layers, the first of which 22a is adhered to the layer of polyethylene material 18 and the second of which 22b is adhered to the first layer 22a but only at opposite edge portions of the web 26a and 26b. In a central portion of the web 28 the second layer 22b is not adhered to the first layer 22a. Thus, by stamping out foil liners in such a way that approximately half the liner intersects the central portion of the web 28 and the other half intersects one of the two edge portions 26a or 26b it is possible to produce the liner shown in Figure 2 and which comprises a semi-circular pull-tab 24 comprising the non-adhered portion of the second form layer 22b. [0007] Although this foil liner overcomes some of the difficulties of the much smaller tab arrangement shown in Figure 1, both suffer from the disadvantage that it is

[0007] Although this foil liner overcomes some of the difficulties of the much smaller tab arrangement shown in Figure 1, both suffer from the disadvantage that it is possible for a consumer to exert a pulling force on the tab in a direction which is by no means an efficient one if they are attempting to remove the foil from the container opening. This, again, can lead to a perceived unreliability and a lack of consumer confidence when it comes to the initial removal of the foil liner.

[0008] In US Patent No. 4,815,618 there is described a tamper indicating dispenser closure in which a membrane, in the form of a foil seal, overlies and is adhered to the perimeter of an opening in a container. However, rather than having an upper non-stick layer, the form layer of the previous arrangements is omitted and replaced by a plastics disc which is of slightly smaller surface area than the opening overlain by the membrane and which is adhered to the membrane at the same time as the membrane is adhered to the container. A pull-ring is attached to the disc and is arranged so that a pulling force applied to the pull-ring by a consumer is transmitted to an edge of the circular disc irrespective of the direction in which the consumer pulls on the pull-ring provided that that direction is generally away from the membrane. Because the edge of the disc is close to the edge of the membrane, the pulling force is said to ensure that substantially all of the membrane is removed. Nevertheless, it will be noted that the pulling force exerted by the consumer must be sufficient to simultaneously overcome the bond created between the membrane and the container at all points around the perimeter of the opening, a task which may be sufficient to defeat the attempts of certain consumers. Thus there still remains a need for a reliable means for removing foil liners and the like

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which does not prevent their use by the elderly or the infirm or consumers with reduced dexterity.

[0009] According to a first aspect of the present invention there is provided a pull-tab for use in removing a membrane adhered to a container, the container defining an opening having a periphery and the membrane overlying the opening and being adhered to said periphery so as to prevent access to the contents of the container via said opening prior to the removal of the membrane, the pull-tab comprising a peripheral portion having a shape corresponding to at least part of the periphery of said opening and being joined to the membrane in a region approximate said part and a tab portion joined to the peripheral portion, the peripheral portion comprising means to restrict a pulling force applied to the tab portion from being substantially transmitted to all but a first sector of the peripheral portion until such time as the membrane approximate said first sector has been pulled away from the container.

[0010] According to a second aspect of the present invention there is provided a closure assembly in combination with a container, the container defining an opening having a periphery and the closure assembly comprising a membrane and a pull-tab, the pull-tab being as described above.

[0011] A number of embodiments of the present invention will now be described by way of example with reference to the accompanying drawings in which:

Figure 1 is a cross-sectional view of a foil liner of the prior art;

Figure 2 is a perspective view of another foil liner of the prior art;

Figure 3 is a cross-sectional view of a web from which the foil liner of Figure 2 is stamped;

Figure 4 is an exploded view of the components of a closure assembly in accordance with a first embodiment of the present invention;

Figure 5 is an enlarged cross-sectional view of a metallic foil of the closure assembly of Figure 4; Figure 6 is a plan view of a pull-tab of the closure assembly of Figure 4;

Figure 7 is a plan view of a pull-tab in accordance with a second embodiment of the present invention; Figure 8 is an cross-sectional view of a pull-tab in accordance with a third embodiment of the present invention;

Figure 9 is an enlarged cross-sectional view of a line of weakness extending through a peripheral portion of a pull-tab in accordance with a fourth embodiment of the present invention; and

Figure 10 is an enlarged cross-sectional view of a line of weakness extending through a peripheral portion of a pull-tab in accordance with a fifth embodiment of the present invention.

[0012] Referring to Figure 4 there is shown a neck 30 of a container 32. The remainder of the container 32 has

not been shown as its body shape may take any suitable form and may, for example, be of square, rectangular or circular cross-section and may have an integral handle formed as part of the body shape.

[0013] The neck 30 is provided on an external surface with engagement means 34 with which to engage complimentary engagement means (not shown) provided on a cap 36. In the example shown, the engagement means 34 comprise a helical thread configuration but other engagement means may also be provided. At an upper end, the neck 30 terminates in a rim 38 which in turn defines an opening 40. It is via this opening 40 that access is gained to the contents of the container 32 and through which the contents of the container are dispensed.

[0014] A circular disc known as a metallic foil 42 rests on rim 38 so as to overlie and close opening 40. As shown in more detail in Figure 5, the metallic foil 42 comprises an electrically conductive substrate 44 which is coated on each of its opposed surfaces with a respective layer of adhesive 46 and 48. Each of the adhesive layers 46 and 48 serve to bond the electrically conductive substrate 44 to a respective layer of polyethylene material 50 or 52. The result is a laminate structure with the electrically conductive substrate 44 sandwiched between two layers of polyethylene material 50 and 52.

[0015] A moulded plastics pull-tab 54 rests on an upper surface of the metallic foil 42 and is shown in plan view in Figure 6. The pull-tab comprises an annular peripheral portion 56 and, in the example shown, an annular tab portion 58 which is joined to the peripheral portion 56 by a tongue 60. The peripheral portion 56 is sized so as to overlie the rim 38 of the container 32 and accordingly has an external diameter substantially the same as that of the rim. The internal diameter of the peripheral portion 56 is substantially the same or slightly smaller than the diameter of the opening 40.

[0016] The tab portion 58 is sized so as to be as large as possible while at the same time capable of being received within the peripheral portion 56. Thus the external diameter of the tab portion 58 is slightly smaller than the internal diameter of the peripheral portion 56 thereby defining a near annular space 62 between the two in plan view. The internal diameter of the tab portion 58 is selected so as to allow the insertion of a finger of a user. [0017] In order to further facilitate this process, the pull-tab is moulded so that in its natural or relaxed state the tab portion 58 and tongue 60 occupy a plane that subtends an acute angle to that occupied by the peripheral portion 56. As a result, at a position opposite the tongue 60, the tab portion 58 is raised in height with respect to the peripheral portion 56 by a distance 64 and this enables the tab portion 58 to be grasped more easily and reliably by a user. Nevertheless, the tab portion 58 is joined to the peripheral portion 56 in a manner which is sufficiently flexible so that, when constrained, the tab portion 58 can occupy substantially the same plane as the peripheral portion 56 and sufficiently resilient to en-

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able the tab portion 58 to return to its natural or relaxed state once that constraint has been removed.

[0018] In contrast to the tab portion 58, the peripheral portion 56 is provided with a plurality of lines of weakness which extend in a direction generally transverse to the tongue 60. In the embodiment shown, the lines of weakness take the form of a series of mutually spaced parallel grooves 66-72 which serve to divide the peripheral portion 56 into a number of sectors 74-82. In this regard it will be noted that grooves 66a and 66b are colinear and so in what follows will be referred to simply as groove 66. The same is also true of grooves 68a and 68b which will henceforth be referred to simply as groove 68 and is also true of grooves 70a and 70b which will henceforth be referred to as groove 70. Grooves 72a and 72b will henceforth be referred to simply as groove 72.

[0019] As can be seen from Figure 4, each of the grooves 66-72 is of V-shaped cross-section and is defined, in the case of groove 66, by mutually inclined surfaces 84 and 86.

[0020] In addition the pull-tab 54 is provided with a plurality of circumferentially spaced flanges 88 which project radially outwardly from the peripheral portion 56.

[0021] A cap 36 is also provided and comprises a circular top 90 which merges at a radially outer edge with a depending annular skirt 92. The annular skirt 92 is sized so as to fit over the neck 30 and, as stated previously, is provided on its inner surface with complimentary engagement means (not shown) for repeated and releasable engagement with the engagement means 34 provided on the container 32.

[0022] In order to assemble the components shown in Figure 4, the pull-tab 54 and metallic foil 42 are first received within the annular skirt 92 of the cap 36 with the pull-tab closest to the underside of the top 90. The purpose of the radially outwardly projecting flanges 88 provided on the peripheral portion 56 is to engage the inner surface of the annular skirt 92 above the complimentary engagement means and so retain the pull-tab 54 within the cap 36. Similar elements may also be provided on the metallic foil 42 to ensure that it too is retained within the cap 36. Alternatively, the metallic foil 42 may simply be formed oversize with an external diameter that is sufficient to retain the metallic foil within the annular skirt 92 above the complimentary engagement means.

[0023] Once the container 32 has been filled with the desired contents, the cap 36, together with the metallic foil 42 and the pull-tab 54, is offered up to the neck 30. Depending on the nature of the respective engagement means provided on the container and cap, the cap 36 is then pushed, rotated or otherwise applied to the neck 30 until such time as the metallic foil 42 is pressed against the rim 38 of the opening 40 and the peripheral portion 56 of the pull-tab 54 is pressed against the metallic foil 42. In this condition the assembled cap and neck are exposed to a time varying magnetic field which

gives rise to eddy currents within the electrically conductive substrate 44 and the generation of heat. This heat in turn melts the respective polyethylene layers 50 and 52 and bonds the metallic foil both to the rim 38 of the container 32 and also to the peripheral portion 56 of the pull-tab 54. In so doing the bonding components, comprising the respective layers of adhesive 46 and 48 and the respective layers of polyethylene material 50 and 52, are selected so as to ensure that a permanent bond is established between the metallic foil 42 and the peripheral portion 56 of the pull-tab 54 while a weaker, peelable bond is established between the metallic foil and the container 32. At the same time and in contrast to the peripheral portion 56, the tab portion 58 is not bonded to the metallic foil 42, either because it is not held in intimate contact with the metallic foil by virtue of its tendency to occupy a different plane from that of the peripheral portion or else, in an alternative embodiment, because the metallic foil 42 is provided on its upper surface with a non-stick form layer which underlies the tab portion 58 but not the peripheral portion 56.

[0024] In use the metallic foil 42 serves to maintain the freshness of the product packaged within the container and serves to provide the consumer with an indication that the container has not been subject to unauthorised tampering. Nevertheless, in order to dispense the contents of the container, the metallic foil must first be removed and discarded. This is achieved by unscrewing or otherwise disengaging the cap 36 from the container 32 so as to expose the pull-tab 54. The user then inserts a finger through the central aperture of the tab portion 58. In so doing the user is helped by the resilience of the pull-tab 54 which, once the cap 36 has been removed, is sufficient to cause the tab portion 58 to return to its natural or relaxed state in which it projects upwardly from the peripheral portion 56 and away from the metallic foil 42. The user then pulls on the pull-tab and, irrespective of the direction in which that pulling force is applied, the pulling force is nonetheless transmitted by the tongue 60 to the peripheral portion 56. However, rather than the pulling force being dissipated around the circumference of the peripheral portion 56 in an attempt to simultaneously overcome the bond between the metallic foil 42 and the container 32 at all points around the rim 38, because of the grooves 66-72, the pulling force applied by the user and transmitted by the tongue 60 is applied substantially only in the first sector 74 bounded by groove 66. As a result, in order to begin to peel the metallic foil 42 away from the rim 38 it is necessary only for the pulling force applied by the user to be sufficient to overcome the bond between the metallic foil 42 and the container 32 over the much smaller surface area of the first sector 74. Only once the metallic foil underlying this sector has been pulled away does the peripheral portion 56 hinge about groove 66, bringing mutually inclined surfaces 84 and 86 into abutting relationship so that the pulling force applied by the user is then transmitted to the second sector 76 bounded by

grooves 66 and 68. Again, because the pulling force applied by the user is restricted to overcoming the bond between the metallic foil 42 and the container 32 only in the region defined by the second sector, the pulling force need not be nearly so great as if it were attempting to overcome the strength of the same bond at all points around the rim. Thus, as the process repeats, a very much reduced pulling force is sufficient to overcome the bond between the metallic foil 42 and the container 32 in each of a succession of sectors before that pulling force is transmitted to the next adjacent sector. As a result the metallic foil 42 may be easily and reliably peeled away from the rim 38 and subsequently discarded together with the pull-tab 54.

[0025] With the removal of the metallic foil 42, the opening 40 is exposed and the contents of the container may be dispensed as desired. Should it be necessary to re-close the container 32, the cap 36 may simply be presented to the neck 30 and applied as necessary depending upon the nature of the engagement means 34. The cap 36 may be disengaged and reapplied to the container 32 as many times as necessary.

[0026] Although the peripheral portion 56 of the pull-tab 54 has been described as annular it will be apparent that the peripheral portion could have any shape although it is preferable that the shape correspond, at least approximately, to that of the periphery of the underlying opening 40. Nevertheless, the peripheral portion 56 of the pull-tab 54 may have a shape selected from the list comprising circular, elliptical, oval, triangular, square, rectangular, pentagonal and hexagonal.

[0027] Likewise, although the peripheral portion 56 has been described as having a shape corresponding to the periphery of the opening in its entirety (in other words that the peripheral portion has a shape which, like the rim 38, is closed), it will also be apparent that the peripheral portion may have a shape corresponding to only a part of the periphery of the opening. For example, where the opening is circular and is surrounded by an annular rim as in the example shown in Figure 4, the peripheral portion 56 of the pull-tab 54 may simply comprise a part annular shape as shown, for example, in Figure 7. Such an arrangement would still permit the metallic foil 42 to be pulled away from the underlying rim 38 using a significantly reduced pulling force.

[0028] It will also be appreciated that the external dimensions of the peripheral portion 56 may, if so desired, be other than substantially the same as that of the rim 38. In particular, the external dimensions may be greater than that of the rim thereby leading to the possibility of the peripheral portion being joined to the metallic foil at a position outwardly of the periphery of the opening. Conversely, the external dimensions of the peripheral portion may be less than that of the rim leading to the peripheral portion no longer overlying the rim but instead being joined to the metallic foil at a position inwardly of the periphery of the opening. Similarly, the internal dimensions of the peripheral portion 56 may, if so desired,

be substantially less than the opening 40 although this may have implications for the size and the shape of the tab portion 58.

[0029] Likewise, although the tab portion 58 has been described as a pull-ring, it need not necessarily be of annular shape provided that it may be readily gripped by a user. Thus, the tab portion 58 may comprise a tab of any shape, either with or without a central aperture, and may, for example, comprise a T-shape to facilitate easy gripping by a user. Likewise, although the tab portion 58 has been described as being of a size so as to be capable of being received within the peripheral portion 56 so that both the tab portion and the peripheral portion can, when so constrained, occupy substantially the same plane, this need not necessarily be the case. In particular, the tab portion may, if so desired, overlap the peripheral portion. This will, however, have an adverse affect on the overall height of the pull-tab 54 although, depending upon the nature of the cap 36 and the design constraints imposed upon the closure as a whole, this may be an acceptable trade off for a tab of increased surface area.

[0030] It will also be appreciated that although the tab portion 58 has been described as moulded so that in its natural or relaxed state it occupies a plane that subtends an angle to that occupied by the peripheral portion 56, once again this need not necessarily be the case. As shown in Figure 8, the tab portion 58 may alternatively be moulded so as to occupy a plane substantially parallel to that of the peripheral portion 56. Whilst the plane of the tab portion 58 may be coplanar with that of the peripheral portion 56, the tab portion is nevertheless preferably raised with respect to the peripheral portion so as to facilitate the tab portion being grasped more easily and reliably by a user. Of course, the tab portion 58 may be moulded so as to occupy a plurality of planes, one or more of which may be inclined to that occupied by the peripheral portion while others may be parallel to the peripheral portion.

[0031] Although the lines of weakness have been described as grooves, again it will be apparent that this need not be the case. In particular, the lines of weakness may instead comprise webs of material of reduced thickness or a series of apertures, either blind or representing through-holes, always provided, of course, that the lines of weakness are not so weak as to become frangible when a typical pulling force is applied to the pull-tab by a user. Where the lines of weakness are present in the form of grooves, it will be apparent that the grooves may be of any convenient shape and may, for example, be U-shaped or straight sided as shown in Figures 9 and

[0032] Once again, although the lines of weakness have been described as extending parallel to each other, this need not necessarily be the case as the lines of weakness may, in the case of an annular or part annular peripheral portion overlying an annular rim, extend in a generally radial direction. However, it is preferable that

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the lines of weakness extend in a direction transverse to the direction in which the pulling force applied to the tab portion is transmitted to the peripheral portion. Most preferably of all the lines of weakness extend in a direction substantially perpendicular to the direction in which a pulling force applied to the tab portion is transmitted to the peripheral portion.

[0033] In the embodiment described with reference to Figures 4 and 5 a plurality of lines of weakness are shown which are equally spaced apart such that the perpendicular distance between groove 66 and groove 68 is substantially the same as that between groove 68 and groove 70 and between groove 70 and groove 72. However, this need not necessarily be the case. In another arrangement the lines of weakness may be arranged so that each of the sectors defined by the lines of weakness are of equal surface area or overlie a bond between the metallic foil and the container which is of equal length. In this way the strength of the bond to be overcome between the metallic foil 42 and the container 32 will be substantially the same for each sector. It will, however, mean that the lines of weakness will be spaced closer together at a side of the peripheral portion close to the tongue 60 and will be spaced further apart in a region spaced 90° from the tongue. In any event, the tongue 60 is preferably joined to the peripheral portion 56 centrally of the first sector.

[0034] Although the pull-tab 54 has been described as comprising a plurality of lines of weakness, it would nevertheless be advantageous if the peripheral portion 56 comprised only a single line of weakness since this would still serve to divide the peripheral portion into two sectors. As a result the pull force that would need to be applied in order to remove the metallic foil would only need to overcome the bond associated with each sector in turn and not both sectors simultaneously. Nevertheless, the peripheral portion preferably comprises a plurality of lines of weakness so that once the metallic foil approximate the first sector has been pulled away, the pulling force applied to the tab portion is directed to a further sector of the peripheral portion having components on either side of the first sector. Similarly, once the metallic foil adhered to the second sector has been released from its engagement with the underlying portion of the rim, the pulling force applied to the tab portion is directed to a third sector which again comprises two components, each adjacent a respective one of the two components of the second sector.

[0035] As previously described, the metallic foil 42 is preferably adhered to both the rim 38 and the peripheral portion 56 by exposure to a time varying magnetic field. However, the partial melting or softening of the two layers of polyethylene material 50 and 52 represents a heat activated bond that may be achieved in other ways. For example, the two layers of polyethylene material 50 and 52 and the two layers of adhesive 46 and 48 used to adhere the layers of polyethylene material to the electrically conductive substrate 44 may all be replaced by

layers of heat activated adhesives applied to either side of the electrically conductive substrate. What is important in this context is that a permanent bond is provided between the metallic foil 42 and the pull-tab 54 while a peelable or removable bond is provided between the metallic foil 42 and the container 32. In any event, the bonding of the metallic foil 42 to both the container 32 and the pull-tab 54 may be activated by alternate means. For example, the bond may be activated by exposing the cap and neck assembly to microwave radiation or to a time varying electric field.

[0036] Once again whilst the opening 40 has been described as closed by a metallic foil 42, the metallic foil 42 may, in principle, be replaced by any suitable membrane and may, for example, comprise a web of plastics material. Under such circumstances, the pull-tab 54 may be formed integrally with the membrane which may, in turn, be provided with a line of weakness extending around the periphery of the underlying opening 40. This line of weakness may either be in the plane of the membrane or else in the cylindrical surface of the neck 30. [0037] Finally, although the assembly has been described as comprising a cap 36, this is entirely optional as the container may, under certain circumstances, be adequately sealed simply by use of the metallic foil or membrane 42. However, once the metallic foil or membrane has been removed, re-closure of the container would then not be possible.

Claims

 A pull-tab for use in removing a membrane adhered to a container, the container defining an opening having a periphery and the membrane overlying the opening and being adhered to said periphery so as to prevent access to the contents of the container via said opening prior to the removal of the membrane, the pull-tab comprising

a peripheral portion having a shape corresponding to at least a part of the periphery of said opening and being joined to the membrane in a region approximate said part and a tab portion joined to the peripheral portion, the peripheral portion comprising means to restrict a pulling force applied to the tab portion from being substantially transmitted to all but a sector of the peripheral portion until such time as the membrane approximate said sector has been pulled away from the container.

 A pull-tab in accordance with claim 1 wherein the peripheral portion has a shape selected from the list comprising circular, elliptical, oval, triangular, square, rectangular, pentagonal, hexagonal, arcuate, part-circular, part-elliptical, part-oval, part-triangular, part-square, part-rectangular, part-pentagonal and part-hexagonal.

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- 3. A closure assembly in combination with a container, the container defining an opening having a periphery and the closure assembly comprising a membrane and a pull-tab, the membrane overlying the opening and being adhered to said periphery so as to prevent access to the contents of the container via said opening prior to the removal of the membrane, and the pull-tab comprising a peripheral portion having a shape corresponding to at least a part of the periphery of said opening and being joined to the membrane in a region approximate said part and a tab portion joined to the peripheral portion, the peripheral portion comprising means to restrict a pulling force applied to the tab portion from being substantially transmitted to all but a sector of the peripheral portion until such time as the membrane approximate to said sector has been pulled away from the container.
- 4. The combination of claim 3 wherein the container defines an opening having a periphery having a shape selected from the list comprising circular, elliptical, oval, triangular, square, rectangular, pentagonal and hexagonal.
- 5. The invention of any preceding claim wherein the peripheral portion has a shape corresponding to substantially the entire periphery of said opening.
- 6. The invention of any preceding claim wherein the peripheral portion is adapted so as to at least partially overlie the periphery of the opening.
- 7. The invention of any of claims 1 to 5 wherein the peripheral portion is adapted so as to be joined to the membrane at a position outwardly of the periphery of the opening.
- 8. The invention of any of claims 1 to 5 wherein the peripheral portion is adapted so as to be joined to the membrane at a position inwardly of the periphery of the opening.
- **9.** The invention of any preceding claim wherein the tab portion comprises a pull-ring.
- **10.** The invention of any preceding claim wherein the tab portion is sized so as not to overlap the peripheral portion in plan view.
- **11.** The invention of any preceding claim wherein the tab portion is formed integrally with the peripheral portion of plastics material.
- 12. The invention of any preceding claim wherein, in a natural state, the tab portion occupies a different plane from that occupied by the peripheral portion, said planes being mutually spaced at at least one

point on the tab portion.

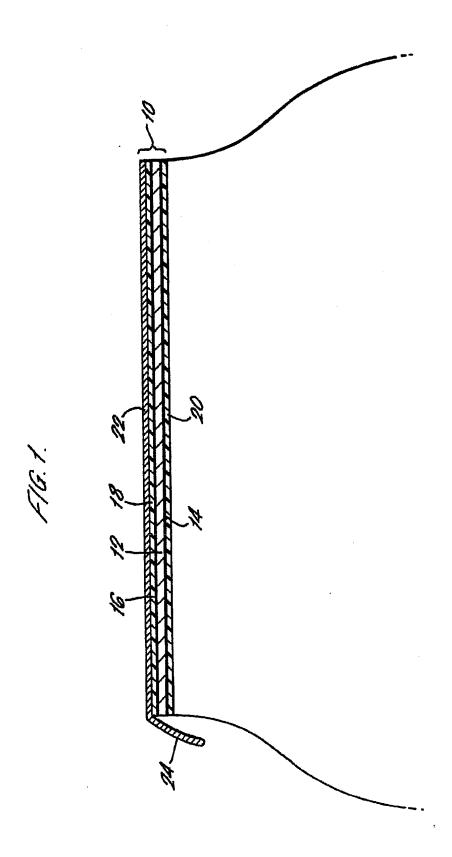
- 13. The invention of any preceding claim wherein, in a natural state, the tab portion occupies a plane that subtends an acute angle with a plane occupied by the peripheral portion.
- 14. The invention of any preceding claim wherein the tab portion is joined to the peripheral portion in a manner which is sufficiently flexible so that, when constrained, the tab portion can occupy substantially the same plane as the peripheral portion and sufficiently resilient to enable the tab portion to return to a natural state once said constraint has been removed in which the tab portion occupies a plane different from that occupied by the peripheral portion.
- 15. The invention of any preceding claim wherein the means to restrict the pulling force applied to the tab portion from being substantially transmitted to all but a sector of the peripheral portion until such time as the membrane approximate said sector has been pulled away from the container comprises one or more lines of weakness that serve to divide the peripheral portion into a plurality of sectors.
- **16.** The invention of claim 15 wherein said lines of weakness comprise one or more grooves.
- 17. The invention of claim 16 wherein the grooves have a cross-sectional shape selected from the list comprising V-shaped, U-shaped or straight-sided-channel-shaped.
- 35 18. The invention of any of claims 15 to 17 wherein the or each line of weakness extends in a direction transverse to the direction in which a pulling force applied to the tab portion is transmitted to the peripheral portion.
 - 19. The invention of any of claims 15 to 18 wherein the or each line of weakness extends in a direction substantially perpendicular to the direction in which a pulling force applied to the tab portion is transmitted to the peripheral portion.
 - 20. The invention of any of claims 15 to 19 and comprising two or more lines of weakness, each line of weakness extending substantially parallel to the other or others.
 - 21. The invention of any of claims 15 to 20 and comprising two or more lines of weakness wherein each line of weakness is equally spaced apart.
 - 22. The invention of any of claims 15 to 20 and comprising two or more lines of weakness wherein the lines of weakness are spaced so that each of the

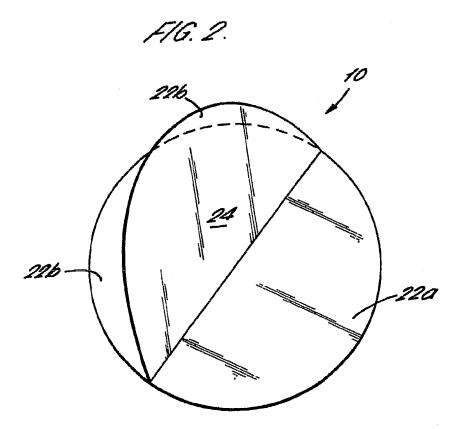
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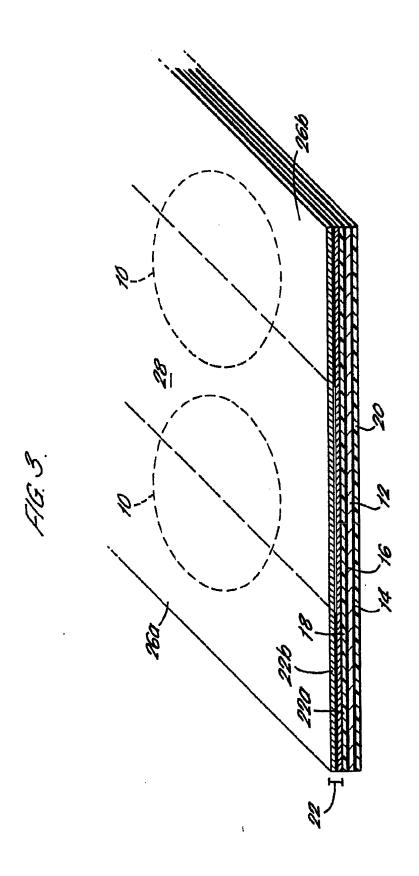
sectors defined thereby are of substantially equal peripheral extent.

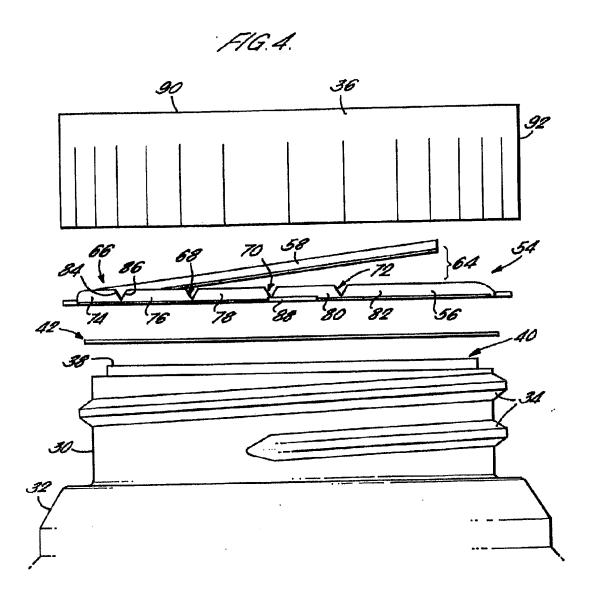
- 23. The invention of any of claims 15 to 22 and comprising two or more lines of weakness wherein the lines of weakness are arranged symmetrically about the direction in which a pulling force applied to the tab portion is transmitted to the peripheral portion.
- 24. The invention of any of claims 15 to 23 wherein the tab portion is joined to the peripheral portion centrally of one of said sectors.
- 25. The invention of any of claims 15 to 24 wherein once said membrane approximate a first sector has been pulled away from the container, the pull-tab is adapted to direct the pulling force applied to the tab portion to a second sector of the peripheral portion, the second sector having components adjacent opposite sides of said first sector.
- 26. The invention of any of claims 15 to 25 wherein once said membrane approximate a first sector has been pulled away from the container, the pull-tab is adapted to direct the pulling force applied to the tab portion to each of the remaining sectors in succession whilst ensuring that the pulling force is not transmitted to a sector until such time as the membrane approximate all of the preceding sectors has been pulled away from the container.
- 27. The invention of any preceding claim wherein the peripheral portion is joined to the membrane by a permanent bond and the membrane is adhered to said periphery by a weaker, peelable bond.
- 28. The invention of any preceding claim wherein the membrane comprises an electrically conductive material and is adhered to the periphery of the opening by a heat activated bond.
- 29. The invention of claim 28 wherein the heat activated bond is activated by exposing the membrane to one of a list comprising microwave radiation, a time varying magnetic field and a time varying electric field.
- **30.** The invention of any preceding claim wherein the membrane comprises a metallic foil.
- **31.** The invention of any of claims 1 to 26 wherein the membrane is formed of plastics material and is formed integrally with the peripheral portion.
- **32.** The invention of claim 31 wherein a line of weakness is provided extending around the periphery of the opening.

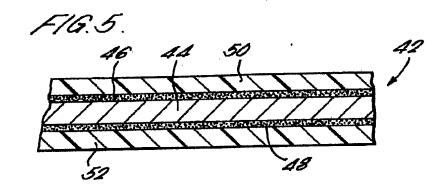
- 33. The invention of any of claims 3 to 32 wherein the closure assembly additionally comprises a cap for repeated and releasable engagement with the container and which, when engaged with the container, overlies the opening and, prior to the removal of the membrane, also overlies both the membrane and the pull-tab.
- **34.** The invention of claim 33 wherein the pull-tab is provided with one or more outwardly directed flanges for engagement with an inner surface of the cap.

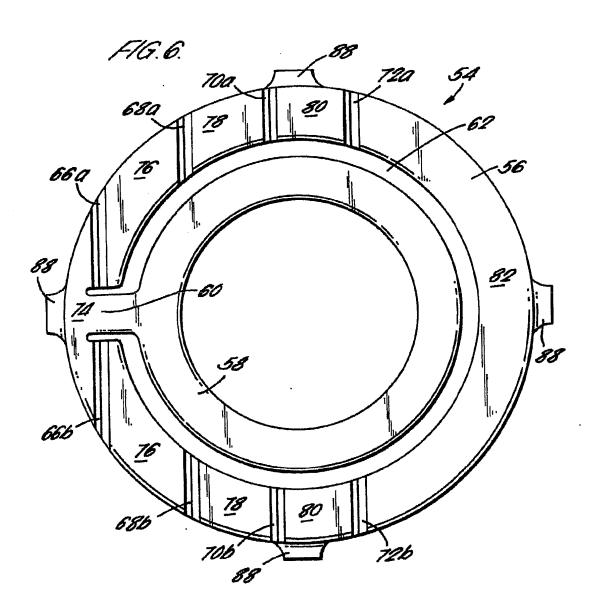


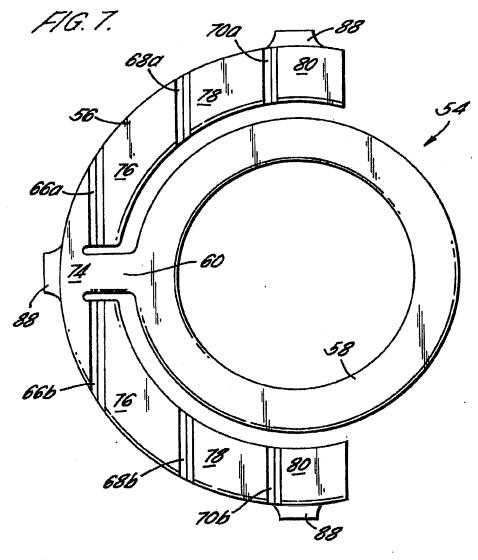


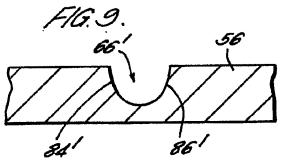


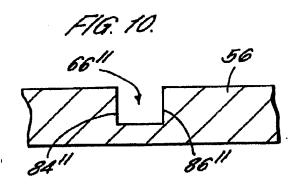


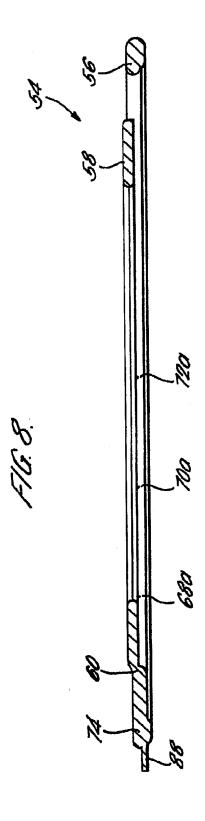














(1) Numéro de publication : 0 425 391 B1

(12)

FASCICULE DE BREVET EUROPEEN

(45) Date de publication du fascicule du brevet : 08.06.94 Bulletin 94/23

(51) Int. CI.⁵: **A61K 35/78,** A23L 1/00,

A61K 7/00

(21) Numéro de dépôt : 90403042.6

(22) Date de dépôt : 26.10.90

- 64) Compositions à base de suc et de protoplastes de végétaux, leur procédé d'obtention et leurs utilisations, notamment dans le domaine de la phytothérapie.
- (30) Priorité: 27.10.89 FR 8914186
- (43) Date de publication de la demande : 02.05.91 Bulletin 91/18
- (45) Mention de la délivrance du brevet : 08.06.94 Bulletin 94/23
- 84 Etats contractants désignés : AT BE CH DE DK ES GB GR IT LI LU NL SE
- 56 Documents cités : EP-A- 420 729 FR-A- 977 029 FR-A- 2 443 265

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Description

La présente invention concerne des compositions à base de protoplastes de végétaux, ainsi que leur procédé d'obtention et leurs utilisations, notamment dans le domaine de la phytothérapie tant par voie interne que par voie externe.

Deux types principaux de produits sont actuellement utilisés, en phytothérapie à savoir

- ceux qui font intervenir la plante entière sèche ou fraîche tels que les plantes séchées pour tisanes ou dispensées en gélules ou comprimés par exemple, et
- les extraits de plantes obtenus à l'état sec tels que les nébulisats, ou sous forme liquide tels que les teintures et les extraits fluides.

Les plantes entières présentent l'inconvénient de posséder une faible concentration en principes actifs mais l'avantage de proposer la totalité de ces derniers.

Les extraits présentent l'inconvénient de ne comporter qu'une partie des composants de la plante mais ont l'avantage de posséder une forte concentration en principes actifs quand ceux-ci sont présents et ce en particulier dans les formes sèches de ces extraits.

La présente invention a précisément pour but de proposer des compositions végétales présentant l'avantage non seulement de comporter l'ensemble des principes actifs de la plante mais également de posséder une très forte concentration en ces principes actifs.

L'invention propose également une méthode particulièrement efficace et simple à mettre en oeuvre pour l'obtention de telles compositions.

L'invention a plus particulièrement pour objet une composition végétale sèche issue d'une plante, cette composition étant exempte des divers constituants des membranes cellulosiques entourant les cellules végétales, en particulier de cellulose, et formée de protoplastes en mélange avec des constituants hydrosolubles appartenant au suc de cette plante, cette composition permettant de reconstituer l'essentiel du suc du végétal dont elle est issue lorsqu'elle est mise en suspension dans de l'eau.

En effet la cellule végétale a la particularité que ne possède pas la cellule animale d'être entourée d'une membrane cellulosique qui représente un poids important dans la plante. Cette membrane ne présente généralement pas d'intérêt thérapeutique à cause de son inertie chimique.

La composition végétale selon l'invention est plus particulièrement caractérisée:

 en ce qu'elle comprend des protoplastes, d'un diamètre compris entre 0,45μm et 100μm, insolubles dans l'eau, ces protoplastes étant eux-mêmes constitués du noyau de la cellule végétale et de cytoplasme cellulaire, en ce que la teneur en protoplastes est d'environ 10 à 20% en poids sec, les 90 à 80% restant étant représentés par des produits hydrosolubles habituellement contenus dans les sucs de plantes, notamment des sucres, des sels minéraux, des protéines etc...

L'invention a également pour objet un procédé d'obtention d'une composition selon l'invention caractérisé en ce qu'il comprend:

- un premier broyage de la plante entière, ou de parties de cette plante (tiges, racines, feuilles etc...) à une température comprise entre -10°C et -40°C, de préférence à environ -25°C,
- un second broyage des fractions de plantes obtenues lors de l'étape de broyage précédente, à une température comprise entre -40°C et -100°C, de préférence à environ -70°C,
- le tamisage des fractions obtenues lors de l'étape de broyage précédente, maintenues à une température comprise entre -40°C et -100°C, à l'aide de tamis de granulométrie variant d'environ 100μm à environ 500μm, suivi de la récupération des fractions passées à travers ce tamis,
- le pressage des fractions récupérées à l'étape précédente, et ramenées à une température de 0°C ± 5°C; les fractions pressées sont ensuite avantageusement soumises au cycle d'opérations suivantes:
 - congélation à une température comprise entre -10°C et -40°C, de préférence à environ -25°C,
 - mise en suspension de ces fractions congelées dans une quantité d'eau approximativement égale à la quantité de liquide obtenu lors de l'étape de pressage précédente,
 - pressage des fractions en suspension dans l'eau et ramenées à une température de $0^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

ce cycle étant répété entre 2 et 6 fois, de préférence 4 fois,

- la filtration des quantités de liquides obtenus lors de chacune des étapes de pressage précédentes, ces quantités étant, le cas échéant, réunies en une solution unique, sur un filtre ayant une porosité de 100μm, et récupération des filtrats (ou du filtrat)
- la concentration des filtrats (ou du filtrat) par une méthode d'élimination de l'eau à froid, notamment par osmose inverse, à la température de 0° C \pm 5° C,
- avantageusement, la congélation des solutions (ou de la solution) concentrées obtenues à l'étape précédente, à une température de -10°C à -40°C, notamment de l'ordre de -25°C, suivie d'une étape de lyophilisation ce qui conduit à l'obtention d'une composition se-

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lon l'une des revendications 1 à 3 sous forme de poudre sèche.

Avantageusement la plante végétale mise en oeuvre dans ce procédé est fraîchement récoltée, c'est-à-dire depuis moins de 24 heures, et est congelée étalée en couches minces en chambre froide à -25°C environ.

Lors de la première étape de broyage du procédé décrit ci-dessus, la plante est coupée grossièrement, notamment à l'aide d'un broyeur à couteaux horizontaux. Cette opération est réalisée à -25°C environ dans l'enceinte de la chambre froide et en régulant la température dans le broyeur avec une addition d'azote liquide.

La seconde étape de broyage du procédé de l'invention, ou encore étape de cryobroyage proprement dit, est avantageusement réalisée en portant la plante, précédemment coupée, à une température de -70°C environ dans un tunnel où l'on introduit de l'azote liquide et dans lequel la plante est acheminée par une vis d'archimède. Les fractions de plante obtenues lors du premier broyage sont ainsi conduites jusqu'à un broyeur à marteaux lui-même refroidi à environ -70°C par de l'azote liquide. Ce second broyage est avantageusement effectué au-dessus d'un tamis de granulométrie variant d'environ 100µm à environ 500µm. Les fractions de plante obtenues lors du second broyage tombent directement dans ce tamis, et l'on récupère les fractions passées à travers le tamis.

Les fractions ainsi récupérées sont portées sans intervention extérieure à la température de 0°C ± 5°C puis l'étape de pressage du procédé de l'invention est avantageusement réalisée dans une presse à vis jusqu'à une pression ne permettant pas d'exclure de liquide de façon significative. Le liquide, ou la suspension, est recueillie et, après passage sur un tamis de porosité 100 µm, est placée en congélation en couches de 2cm dans des plateaux. Conformément au cycle d'opérations décrit dans le procédé de l'invention, les fractions de plante pressées sont recongelées à -25°C par passage dans le même tunnel que celui décrit précédemment par aspersion d'azote liquide. On recommence alors la même opération de pressage que ci-dessus mais après avoir ajouté à la plante une quantité d'eau de préférence purifiée égale à la quantité de liquide exprimé lors du premier pressage.

Cette succession de congélation-décongélationpressage est recommencée en tout quatre fois avec à chaque fois addition d'une quantité d'eau purifiée de préférence égale à la quantité de liquide obtenue lors du pressage précédent.

L'étape de concentration du procédé de l'invention est avantageusement réalisée par osmose inverse, de préférence à froid afin de ne pas dénaturer les composants végétaux contenus dans les liquides obtenus lors des étapes de pressage. L'opération est réalisée à 0°C \pm 5°C par passage permanent des li-

quides sur un échangeur dans lequel circule du fluide réfrigérant. L'osmose est réalisée à environ 40 bars sur une membrane dont le seuil de rétention dans l'eau pure vis-à-vis du chlorure de sodium est de 99,9%.

Le volume final représente environ le dixième du volume initial.

Avantageusement, les suspensions sont toutes congelées en couches minces de 2cm et sont ensuite lyophilisées. La poudre sèche recueillie est ensuite broyée finement et tamisée selon l'usage qui doit en être fait.

Les compositions de l'invention se présentent donc sous forme de solutions, notamment aqueuses, plus ou moins concentrées en constituants végétaux, ou avantageusement sous forme d'un lyophilisat de poudre sèche.

L'étape de lyophilisation du procédé de l'invention est particulièrement avantageuse en ce qu'elle permet d'obtenir une poudre sèche (lyophilisat) dans laquelle l'intégrité des protoplastes est conservée.

A titre illustratif, la teneur en protoplastes d'une composition de l'invention peut être déterminée de la manière suivante:

- mise en suspension dans l'eau d'une quantité aliquote de ladite composition se présentant sous forme de poudre,
- filtration sur un filtre de porosité 0,45µm et récupération de la fraction insoluble dans l'eau qui reste sur le filtre,
- le cas échéant, étude au microscope de la fraction sus-mentionnée afin de vérifier qu'elle est bien essentiellement constituée de protoplastes,
- séchage de la fraction restée sur le filtre, et détermination du poids de cette fraction ainsi séchée par rapport au poids de la poudre de départ.

L'invention a également pour objet des compositions pharmaceutiques ou cosmétiques, comprenant une composition végétale selon l'invention, ou un mélange de plusieurs de ces compositions issues de végétaux différents, le cas échéant en association avec un véhicule physiologiquement acceptable.

A ce titre l'invention concerne plus particulièrement des compositions pharmaceutiques, ou cosmétiques, se présentant sous forme de gélules, ou de comprimés pour l'administration orale, ou encore sous forme de gels, d'émulsions ou de pommages pour un usage topique.

L'invention vise également une méthode de traitement esthétique comprenant l'administration, notamment par voie topique ou orale, d'une composition cosmétique de l'invention.

L'invention concerne également des compositions alimentaires ou diététiques comprenant une (ou plusieurs) composition de l'invention, le cas échéant en association avec un véhicule alimentaire.

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Toute plante dont le suc est susceptible de contenir des constituants actifs dans le domaine de la phytothérapie, de la cosmétologie, ou encore de la diététique (notamment les plantes officinales ou médicinales) est utilisable dans le cadre de la présente invention.

L'invention sera plus particulièrement illustrée à l'aide des exemples de mise en oeuvre du procédé d'obtention de compositions de l'invention qui suivent, étant bien entendu que ces exemples ne sont nullement limitatifs.

- Exemple 1

200kg de parties aériennes fraiches de Equisetum arvense sont récoltés et placés en couches de 5cm environ dans une chambre froide à -25°C. Après 24 heures, la plante (ou drogue) est broyée dans un broyeur à couteaux pour donner des morceaux de 1 à quelques cm. Puis la drogue coupée est admise dans le cryobroyeur et amenée à une température de -70°C. La température du broyeur est régulée à -85°C et le tamis possède une porosité de 350μm.

La poudre congelée ainsi obtenue est laissée à température ambiante jusqu'à ce qu'elle atteigne 4°C. Puis on la presse dans la presse à vis. On obtient alors environ 160kg de suspension.

Ces 160kg de suspension sont osmosés à -2°C jusqu'à obtenir 5kg de rétentat. Le rétentat est ensuite réparti en couches de 2cm d'épaisseur dans des plateaux et placé au congélateur à -25°C.

La plante a été cependant remise au congélateur jusqu'à ce qu'elle revienne à la température de -25°C. On la mélange alors à 160kg d'eau purifiée préalablement refroidie à 4°C et on attend à température ambiante que la plante atteigne à nouveau 4°C. On réalise alors le deuxième pressage. On obtient 180kg de suspension que l'on osmose jusqu'à obtenir 5kg de rétentat placés au congélateur dans les mêmes conditions que précédemment.

On réalise ensuite la même opération encore deux fois. On obtient successivement 140kg et 130kg de suspension et en finale 20kg de rétentat qui sont ensuite filtrés sur filtre (grille) de porosité 100µm, et lyophilisés. Le rendement final en matière sèche lyophilisée est de 12kg.

Etant donné que la plante de départ contenait 84% d'eau le rendement par rapport à la matière sèche de départ est de 37,5%.

La teneur en protoplaste peut être estimée comme étant la fraction de la suspension qui est retenue sur des filtres de porosité 0,45µm et qui passe sur des filtres de porosité 100µm; dans ce cas on peut estimer la teneur en protoplastes à 12% de la matière sèche totale le reste de la matière sèche est représentée par des substances en solution et des substances issues de la destruction mécanique des cellules.

- Exemple 2

200kg de somités fleuries fraîches de Spirea ulmaria sont récoltés et placés en couches de 10cm environ dans une chambre froide à -25°C. Après 16 heures, la drogue est broyée dans un broyeur à couteaux pour donner des morceaux de 1 à quelques cm.

Puis la drogue coupée est admise dans le cryobroyeur et amenée à une température de -80°C. La température du broyeur est régulée à -100°C et le tamis possède une porosité de 450μm.

La poudre congelée ainsi obtenue est laissée à température ambiante jusqu'à ce qu'elle atteigne 4°C. Puis on la presse dans la presse à vis. On obtient alors environ 105kg de suspension.

Ces 105kg de suspension sont osmosés à -1°C jusqu'à obtenir 5kg de rétentat. Le rétentat est ensuite réparti en couches de 2cm d'épaisseur dans des plateaux et placé au congélateur à -25°C.

La plante a été cependant remise au congélateur jusqu'à ce qu'elle revienne à la température de -25°C. On la mélange alors à 105kg d'eau purifiée préalablement refroidie à 4°C et on attend à température ambiante que la plante atteigne à nouveau 4°C. On réalise alors le deuxième pressage. On obtient 155kg de suspension que l'on osmose jusqu'à obtenir 5kg de rétentat placés au congélateur dans les mêmes conditions que précédemment.

On réalise ensuite la même opération encore deux fois. On obtient successivement 120kg et 150kg de suspension et en finale 20kg de rétentat qui sont ensuite filtrés sur filtre (grille) de porosité 100µm, et lyophilisés. Le rendement final en matière sèche lyophilisée est de 9kg.

Etant donné que la plante de départ contenait 78% d'eau le rendement par rapport à la matière sèche de départ est de 20,45%.

La teneur en protoplaste peut être estimée comme étant la fraction de la suspension qui est retenue sur des filtres de porosité 0,45µm et qui passe sur des filtres de porosité 100µm. Dans ce cas on peut estimer la teneur en protoplastes à 17% de la matière sèche totale le reste de la matière sèche est représentée par des substances en solution et des substances issue de la destruction mécanique des cellules.

- Exemple 3

200kg de parties aériennes de Fumaria officinalis fraîches sont récoltés et placés en couches de 5cm environ dans une chambre froide à -25°C. Après 22 heures, la drogue est broyée dans un broyeur à couteaux pour donner des morceaux de 1 à quelques cm.

Puis la drogue coupée est admise dans le cryobroyeur et amenée à une température de -70°C. La température du broyeur est régulée à -90°C et le tamis possède une porosité de 500μm.

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La poudre congelée ainsi obtenue est laissée à température ambiante jusqu'à ce qu'elle atteigne 4°C. Puis on la presse dans la presse à vis. On obtient alors environ 155kg de suspension.

Ces 155kg de suspension sont osmosés à 2°C jusqu'à obtenir 5kg de rétentat. Le rétentat est ensuite réparti en couches de 2cm d'épaisseur dans des plateaux et placés au congélateur à -25°C.

La plante a été cependant remise au congélateur jusqu'à ce qu'elle revienne à la température de -25°C. On la mélange alors à 205kg d'eau purifiée préalablement refroidie à 4°C et on attend à température ambiante que la plante atteigne à nouveau 4°C. On réalise alors le deuxième pressage. On obtient 125kg de suspension que l'on osmose jusqu'à obtenir 5kg de rétentat placés au congélateur dans les mêmes conditions que précédemment.

On réalise ensuite la même opération encore deux fois. On obtient successivement 100kg et 135kg de suspension et en finale 20kg de rétentat qui sont ensuite filtrés sur filtre (grille) de porosité 100µm, et qui sont ensuite lyophilisée. Le rendement final en matière sèche lyophilisée est de 10kg.

Etant donné que la plante de départ contenait 82% d'eau le rendement par rapport à la matière sèche de départ est de 21,77%.

La teneur en protoplaste peut être estimée comme étant la fraction de la suspension qui est retenue sur des filtres de porosité 0,45µm et qui passe sur des filtres de porosité 100µm. Dans ce cas on peut estimer la teneur en protoplastes à 11% de la matière sèche totale le reste de la matière sèche est représentée par des substances en solution et des substances issues de la destruction mécanique des cellules.

Revendications

- Composition végétale sèche issue d'une plante, cette composition étant exempte de cellulose et formée de protoplastes en mélange avec des constituants hydrosolubles appartenant au suc de cette plante, pour reconstituer l'essentiel du suc du végétal dont cette composition est issue lorsqu'elle est mise en suspension dans de l'eau
- Composition selon la revendication 1, caractérisée en ce qu'elle se présente sous la forme d'un lyophilisat.
- Composition végétale selon la revendication 1 ou la revendication 2, caractérisée en ce qu'elle comprend des protoplastes, d'un diamètre compris entre 0,45μm et 100μm, insolubles dans l'eau.
- 4. Composition selon l'une des revendications 1 à

3, caractérisée en ce que la teneur en protoplastes est d'environ 10 à 20% en poids sec.

- 5. Procédé d'obtention d'une composition selon l'une des revendications 1 à 4, caractérisé en ce qu'il comprend:
 - un premier broyage de la plante entière, ou de parties de cette plante, à une température comprise entre -10°C et -40°C, de préférence à environ -25°C,
 - un second broyage des fractions de plantes obtenues lors de l'étape de broyage précédente, à une température comprise entre -40°C et -100°C, de préférence à environ -70°C,
 - le tamisage des fractions obtenues lors de l'étape de broyage précédente, maintenues à une température comprise entre -40°C et -100°C, à l'aide de tamis de granulométrie variant d'environ 100μm à environ 500μm, suivi de la récupération des fractions passées à travers ce tamis,
 - le pressage des fractions récupérées à l'étape précédente, et ramenées à une température de 0°C ± 5°C; les fractions pressées sont ensuite soumises au cycle d'opérations suivantes:
 - congélation à une température comprise entre -10°C et -40°C, de préférence à environ -25°C,
 - mise en suspension de ces fractions congelées dans une quantité d'eau approximativement égale à la quantité de liquide obtenu lors de l'étape de pressaqe précédente,
 - pressage des fractions en suspension dans l'eau et ramenées à une température de 0°C ± 5°C, ce cycle étant répété entre 2 et 6 fois, de préférence 4 fois,
 - la filtration des quantités de liquides obtenus lors de chacune des étapes de pressage précédentes, ces quantités étant, le cas échéant, réunies en une solution unique, sur un filtre ayant une porosité de 100μm, et récupération des filtrats (ou du filtrat)
 - la concentration des filtrats (ou du filtrat) par une méthode d'élimination de l'eau à froid, notamment par osmose inverse, à la température de 0°C ± 5°C,
 - avantageusement, la congélation des solutions (ou de la solution) concentrées obtenues à l'étape précédente, à une température de -10°C à -40°C, notamment de l'ordre de -25°C, suivie d'une étape de lyophilisation ce qui conduit à l'obtention d'une composition selon l'une des revendications 1 à 4 sous forme de poudre sèche.

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- 6. Composition pharmaceutique caractérisée en ce qu'elle comprend une composition selon l'une des revendications 1 à 4 en association avec un véhicule physiologiquement acceptable.
- Composition cosmétique caractérisée en ce qu'elle comprend une composition selon l'une des revendications 1 à 4 en association avec un véhicule physiologiquement acceptable.
- Composition selon la revendication 6 ou la revendication 7, caractérisée en ce qu'elle se présente sous forme de gélules, comprimés, gel, émulsion ou pommade.
- Composition alimentaire ou diététique, caractérisée en ce qu'elle comprend une composition selon l'une des revendications 1 à 4.

Patentansprüche

- Trockene, pflanzliche Zusammensetzung pflanzlichen Ursprungs, die frei von Cellulose ist und von Protoplasten in Mischung mit wasserlöslichen, zu dem Saft der Pflanze gehörenden Bestandteilen gebildet ist, zur Rekonstitution des Wesentlichen des dieser Zusammensetzung zugrundeliegenden Pflanzensaftes bei Suspension derselben in Wasser.
- Zusammensetzung nach Anspruch 1, dadurch gekennzeichnet, daß die in Form eines Lyophilisats vorliegt.
- 4. Zusammensetzung nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß der Gehalt an Protoplasten etwa 10 bis 20 %, bezogen auf Trockengewicht, beträgt.
- Verfahren zur Herstellung einer Zusammensetzung nach einem der Ansprüche 1 bis 4, gekennzeichnet durch die Schritte
 - erstes Zerkleinern der gesamten Pflanze oder von Teilen dieser Pflanze bei einer Temperatur zwischen -10°C und -40°C, vorzugsweise bei etwa -25°C,
 - zweites Zerkleinern der erhaltenen Pflanzenfraktionen im Anschluß an die vorherige Zerkleinerungsstufe bei einer Temperatur zwischen -40°C und -100°C, vorzugsweise bei etwa -70°C,
 - Sieben der erhaltenen Fraktionen nach der

- vorhergehenden Zerkleinerungsstufe, wobei diese bei einer Temperatur zwischen -40°C und -100°C gehalten werden, mit Hilfe von verschiedenen Sieben für die Granulometrie von etwa 100μm bis etwa 500μm, gefolgt von der Sammlung von durch das Sieb gehenden Fraktionen,
- Pressen der in der vorherigen Stufe gewonnenen und auf eine Temperatur von 0°C ± 5°C gebrachten Fraktionen; wobei die geßrepten Fraktionen anschließend einen Zyklus der folgenden Maßnahmen unterworfen werden:
 - . Einfrieren auf eine Temperatur zwischen -10°C und -40°C, vorzugsweise von etwa -25°C,
 - Suspendierung dieser eingefrorenen Fraktionen in einer Wassermenge, die etwa gleich der in der vorherigen Stufe des Pressens erhaltenen Flüssigkeitsmenge ist,
 - Pressen der in Wasser suspendierten und auf eine Temperatur von 0°C ± 5°C gebrachten Fraktionen, wobei dieser Zyklus zwischen 2 und 6, vorzugsweise 4 mal wieder holt wird,
- Filtration der nach einer jeden der vorherigen Stufen erhaltenen Flüssigkeitsmengen, wobei diese Mengen gegebenenfalls zu einer einzigen Lösung vereint werden, durch einen Filter mit einer Porosität von 100µm, und Sammlung des Filtrats bzw. der Filtrate.
- Konzentration des Filtrats bzw. der Filtrate mittels eines Verfahrens zum Ausschluß von Wasser in der Kälte, insbesondere der Umkehrosmose, bei einer Temperatur von 0°C bis ± 5°C,
- vorzugsweise Einfrieren der in der vorherigen Stufe erhaltenen, konzentrierten Lösung(en) bei einer Temperatur von -10°C bis -40°C, insbesondere in der Größenordnung von -25°C, gefolgt von einer Stufe der Lyophilisation, wodurch eine Zusammensetzung gemäß einem der Ansprüche 1 bis 4 in Form eines trockenen Pulvers erhalten wird.
- 6. Pharmazeutische Zusammensetzung, dadurch gekennzeichnet daß sie eine Zusammensetzung gemäß einem der Ansprüche 1 bis 4 zusammen mit einem physiologisch verträglichen Träger enthält.
 - 7. Kosmetische Zusammensetzung, dadurch gekennzeichnet, daß sie eine Zusammensetzung gemäß einem der Ansprüche 1 bis 4 zusammen mit einem physiologisch verträglichen Träger enthält.

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- Zusammensetzung nach Anspruch 6 oder 7, dadurch gekennzeichnet, daß sie in Form von Kapseln, Tabletten, Gelen, Emulsionen oder Salben vorliegt.
- Ernährungszusammensetzung oder diätetische Zusammensetzung, dadurch gekennzeichnet, daß sie eine Zusammensetzung gemäß einem der Ansprüche 1 bis 4 enthält.

Claims

- Dried plant composition derived from a plant, this
 composition being free of cellulose and made up
 of protoplasts in the form of a mixture with watersoluble constituents belonging to the juice of this
 plant, for reconstituting the bulk of the plant juice
 from which this composition is derived when the
 said composition is suspended in water.
- Composition according to Claim 1, characterized in that it is provided in the form of a freeze-dried product.
- Plant composition according to Claim 1 or Claim 2, characterized in that it comprises protoplasts, with a diameter of between 0.45 μm and 100 μm, which are insoluble in water.
- Composition according to one of Claims 1 to 3, characterized in that the protoplast content is about 10 to 20% on dry weight basis.
- 5. Process for producing a composition according to one of Claims 1 to 4, characterized in that it comprises:
 - a first grinding of the whole plant, or of parts of this plant, at a temperature of between -10°C and -40°C, preferably at about -25°C.
 - a second grinding of the plant fractions obtained during the preceding grinding step, at a temperature of between -40°C and -100°C, preferably at about -70°C,
 - sieving the fractions obtained during the preceding grinding step, maintained at a temperature of between -40°C and -100°C, by means of a sieve offering a particle size ranging from about 100 μm to about 500 μm, followed by the recovery of the fractions which passed through this sieve,
 - pressing the fractions recovered in the preceding step and adjusted to a temperature of 0°C±5°C; the pressed fractions are then subjected to the following cycle of operations:
 - . freezing at a temperature of between

- -10°C and -40°C, preferably at about -25°C,
- suspending these frozen fractions in a quantity of water approximately equal to the quantity of liquid obtained during the preceding pressing step,
- pressing the fractions suspended in water and adjusted to a temperature of 0°C ± 5°C, this cycle being repeated between 2 and 6 times, preferably 4 times,
- filtering the quantities of liquid obtained during each of the preceding pressing steps, these quantities being, where appropriate, pooled into a single solution, on a filter having a porosity of 100 μm, and recovering the filtrates (or the filtrate)
- concentrating the filtrates (or the filtrate) by a method for removing water at cold temperature, especially by reverse osmosis, at the temperature of 0°C ± 5°C,
- advantageously, freezing the concentrated solutions (or solution) obtained in the preceding step, at a temperature of -10°C to -40°C, especially of the order of -25°C, followed by a freeze-drying step, thereby resulting in the production of a composition according to one of Claims 1 to 4 in the form of a dry powder.
- 6. Pharmaceutical composition characterized in that it comprises a composition according to one of Claims 1 to 4, combined with a physiologically acceptable vehicle.
- Cosmetic composition characterized in that it comprises a composition according to one of Claims 1 to 4, combined with a physiologically acceptable vehicle.
- Composition according to Claim 6 or Claim 7, characterized in that it is provided in the form of hard gelatin capsules, tablets, gel, emulsion or ointment.
- Food or dietary composition, characterized in that it comprises a composition according to one of Claims 1 to 4.

(11) Nº de publication :

2 439 139

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PARIS

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DEMANDE DE BREVET D'INVENTION

N° 79 26345 (21) **64**) Double couvercle de sûreté avec sécurité pour les enfants. (51) Classification internationale. (Int. Cl 3) B 65 D 41/16, 55/02, 83/14. Date de dépôt 19 octobre 1979. Priorité revendiquée : Demande de brevet déposée en République Fédérale d'Allemagne le 21 octobre 1978, n. P 28 45 966.7. (41) Date de la mise à la disposition du B.O.P.I. - «Listes» n. 20 du 16-5-1980. public de la demande (71) PETER KWASNY KG chemisch - technische Erzeugnisse, résidant en Déposant : République Fédérale d'Allemagne. Peter Kwasny. (72)Invention de : (73) Titulaire: Idem (71) Mandataire : Office méditerranéen des Brevets d'Invention, Cabinet Hautier, (74)

24, rue Masséna, 06000 Nice.

L'invention concerne un couvercle double de sûreté en matière plastique élastique avec sécurité pour les enfants, pour bombes aérosols, avec une enveloppe extérieure qui repose sur la bordure du boîtier et une enveloppe intérieure dont le bord 5 rabattu vers l'intérieur s'engage en dessous de la cloche de soupape, cette enveloppe intérieure étant constituée de deux segments diamétralement opposés et disposés à une certaine distance l'un en regard de l'autre.

Dans un couvercle de sûreté de ce genre, connu par la 10 description du modèle d'utilité déposé sous le n° 77 12650, on peut introduire à travers une fenêtre ménagée dans l'enveloppe extérieure du couvercle, la lame d'un tourvevis dans la fente formée entre les segments de l'enveloppe intérieure du couvercle, tourner la lame du tournevis, élargir ainsi la fente et 15 repousser ainsi vers l'extérieur, dans le sens radial, le bord rabattu vers l'intérieur des segments de la cloche de soupape de manière à libérer le couvercle enclenchable. Les petits enfants n'ont pas encore l'idée d'une telle utilisation d'un tournevis et c'est ce qui constitue la sécurité pour les 20 enfants qui empêche une manoeuvre intempestive de la soupape à pression recouverte par le couvercle de sécurité et disposée dans la cloche de soupape à l'extrémité supérieure du boîtier atomiseur de laque.

Cependant, l'utilisateur qualifié ne dispose pas tou-25 jours d'un tournevis ou de quelque outil analogue de sorte que la possibilité d'emploi d'une bombe de laque ainsi équipée s'en trouve considérablement réduite.

L'objet de la présente invention est de ce fait, de perfectionner un double couvercle de sécurité du genre mentionné 30 dans le préambule, de manière à ce que celui-ci puisse être déverrouillé sans recours à un outil et que malgrè cela, les petits en fants ne puissent pas effectuer ce déverrouillage.

Selon la présente invention, il est proposé pour cela que les deux segments soient réunis par des éléments en forme 35 de coin, moulés avec eux d'une seule pièce, avec une structure en forme de toit, orientés vers l'enveloppe extérieure et liés de préférence à celle-ci le long de leur ligne de faîte.

Si l'on comprime le couvercle en pressant son enveloppe extérieure entre le pouce et l'index dans la région des élé-

ments en forme de coin, l'enveloppe extérieure se déforme et prend une forme ovale et les pièces en forme de coin de l'enveloppe intérieure transmettent ce mouvement aux segments de celle-ci qui s'écartent l'un de l'autre, leurs bords rabattus 5 vers l'intérieur se dégageant de la bordure de la cloche de soupape et il est alors possible de retirer le couvercle. Un petit enfant n'est pas capable de saisir ces relations, il ne peut pas non plus, avec le diamètre des couvercles des bombes habituelles du commerce, saisir ce couvercle entre le pouce et l'index, tout au moins pas avec la force nécessaire pour en provoquer la déformation et c'est en ceci que consiste la sécurité pour les enfants, revendiquée selon la présente invention.

Selon des extensions de l'invention, l'enveloppe extérieure peut comporter des zones en retrait ou une épaisseur de paroi réduite dans la zone qui entoure les éléments de la paroi intérieure. Par compression, on obtient alors une forme ovale aplatie, plus prononcée, un effet de levier augmenté en conséquence s'exerce sur les segments de l'enveloppe intérieure par l'intermédiaire des éléments en forme de coin et les segments de l'enveloppe intérieure s'écartent alors davantage.

De plus, on peut prévoir sur l'enveloppe extérieure un striage sur chacune des parties de la périphérie qui correspondent aux éléments en forme de coin. Ceci empêche que les doigts de l'utilisateur ne dérapent sur l'enveloppe du couvercle lorsqu'on comprime celui-ci.

On explique ci-dessous l'invention plus en détail avec l'exemple de réalisation représenté par le dessin dont les différentes figures représentent respectivement :

- 30 - Figure 1 : une vue de profil.
 - Figure 2 : une coupe radiale de celle-ci.
 - Figure 3: une coupe axiale selon le plan B/B de la figure 2.
 - Figure 4: une autre coupe axiale selon le plan A/A de la figure 2 avec le couvercle emboîté sur la bombe.
- Figure 5: une vue en perspective illustrant le 35 maniement du couvercle.

Le couvercle double de protection est moulé par injection, en une seule pièce en matière plastique élastique, il

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est constitué d'une enveloppe extérieure (1) et d'une enveloppe intérieure qui se compose de deux segments 2, disposés diamétralement en regard et à une certaine distance l'une de l'autre. Ces derniers sont réunis par des éléments en forme de 5 coin 4 ayant une structure en forme de toit, orientés vers l'enveloppe extérieure 1 et réunis à celle-ci le long de leurs lignes de faîte 3. Dans la zone des lignes de faîte 3, sur la surface extérieure de l'enveloppe extérieure 1, des striages 5 sont prévus, diamétralement opposés par conséquent l'un à l'autre.

Le couvercle double de sécurité repose de la manière connue en soi sur une bombe de pulvérisation 6 également connue, telles que celles qui sont représentées en partie dans les figures 4 et 5.

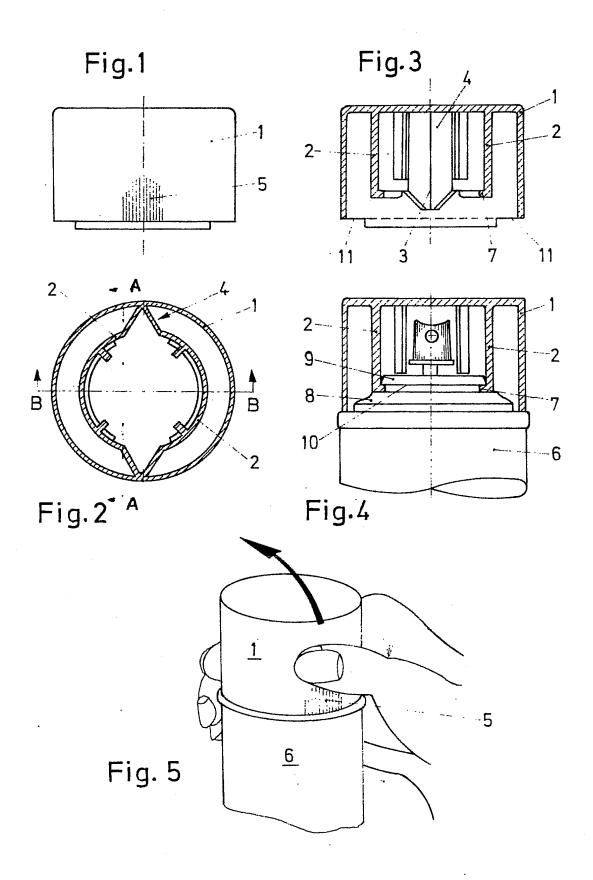
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Le rebord 7 des parties intérieures 2 de l'enveloppe 15 intérieure est alors engagé en dessous de la cloche de soupape 8 dans l'ourlet 10 formé à la jonction de celle-ci avec le disque de soupape 10, ceci apparaissant le plus clairement dans la figure 4.

20 Si maintenant on comprime l'un contre l'autre, comme on l'a représenté dans la figure 5, les deux striages 5 prévus sur l'enveloppe extérieure 1 du couvercle, celui-ci se déforme et prend une forme ovale, ce qui est encore favorisé par le fait que dans la partie qui entoure l'enveloppe intérieure 2, l'enveloppe extérieure 1 comporte des parties en retrait 11 25 que l'on voit mieux sur les figures 1 et 3.

REVENDICATIONS

- 1. Couvercle de sécurité double en matière plastique élastique, avec sécurité pour la protection des enfants, pour les bombes aérosols, avec une enveloppe extérieure reposant sur la bordure du boîtier de la bombe et avec une enveloppe intérieure dont le bord rabattu vers l'intérieur s'engage en dessous de la cloche de soupape, cette enveloppe intérieure étant constituée de deux segments disposés diamétralement en regard et à une certaine distance l'un par rapport à l'autre, caractérisé par le fait que les segments (2) sont réunis entre eux au moyen d'éléments en forme de coin (4) venus de moulage en une seule pièce en forme de toit, orientés vers l'enveloppe extérieure (1) et liés de préférence à celle-ci le long de leurs lignes de faîte (3).
- Couvercle de sécurité double selon la revendication
 1, caractérisé par le fait que l'enveloppe extérieure (1) comporte des parties en retrait (11) et/ou une épaisseur de paroi réduite dans la partie entourant les segments intérieurs (2).
- 3. Couvercle de sécurité double selon l'une quelconque des revendications 1 ou 2, caractérisé par le fait que dans chacune des zones périphériques qui correspondent aux pièces en forme de coin (4), l'enveloppe extérieure (1) comporte à l'extérieur un striage (5).



UK Patent Application (19) GB (11) 2 148 235 A

(43) Application published 30 May 1985

- (21) Application No 8426387
- (22) Date of filing 18 Oct 1984
- (30) Priority data

(31) 543698	(32) 20 Oct 1983	(33) US
543692	20 Oct 1983	
543699	20 Oct 1983	
557306	2 Dec 1983	
557502	2 Dec 1983	
557500	2 Dec 1983	
641663	17 Aug 1984	

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- (51) INT CL⁴
 A61J 3/07
- (52) Domestic classification B8C A B5A 1R314C12 1R314C1S 1R324 1R409 20T14 20T16 20T17 20T1 20T3 C
- (56) Documents cited GB 1496737

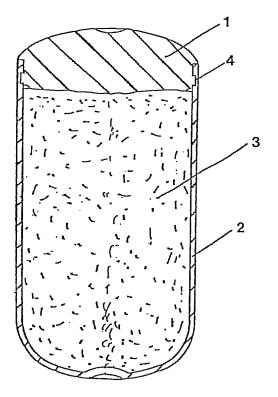
GB 1355324

(58) Field of search B5A B8C

(54) Die pressure moulded capsules

(57) A die pressure moulded article is made from a hydrophilic polymer, a mixture of such polymers, a starch, a starch derivative, or mixtures thereof. The articles may be injection moulded capsules having a joinable cap part (1) and body part (2). The cap part (1) may be die-moulded as a stopper directly onto the open end of the body part (2) after the body has been filled so as to seal the contents (3). Alternatively, each of the cap part (1) and body part (2) may have, in the side wall area, adjacent to its open end, at least one locking means (4).





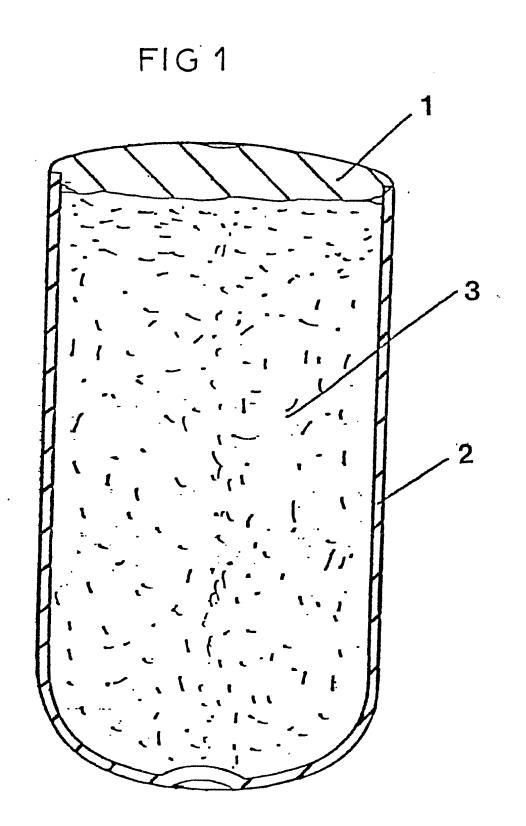
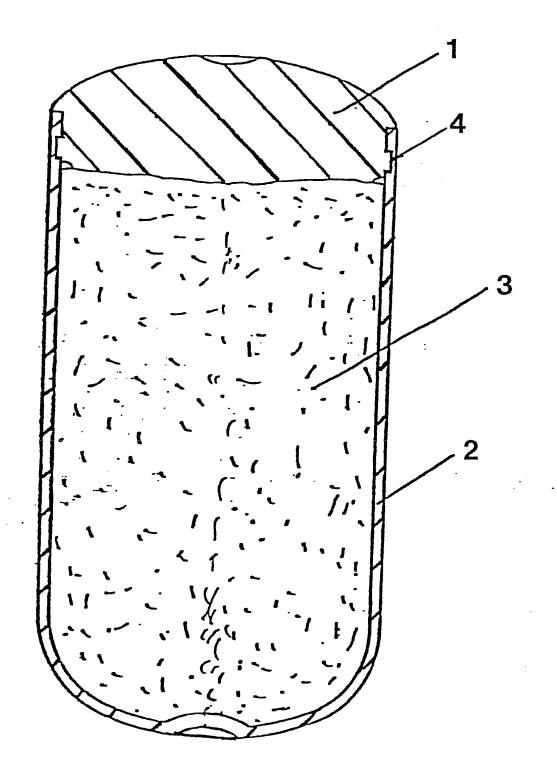
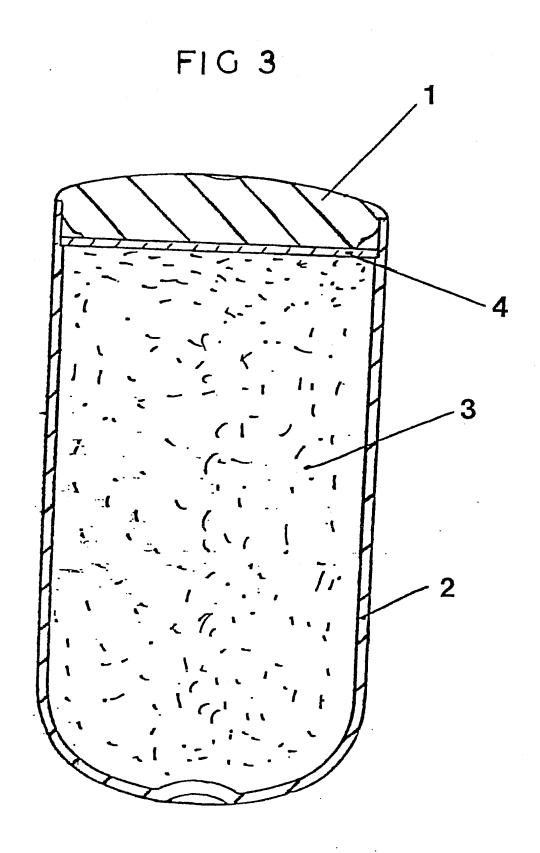
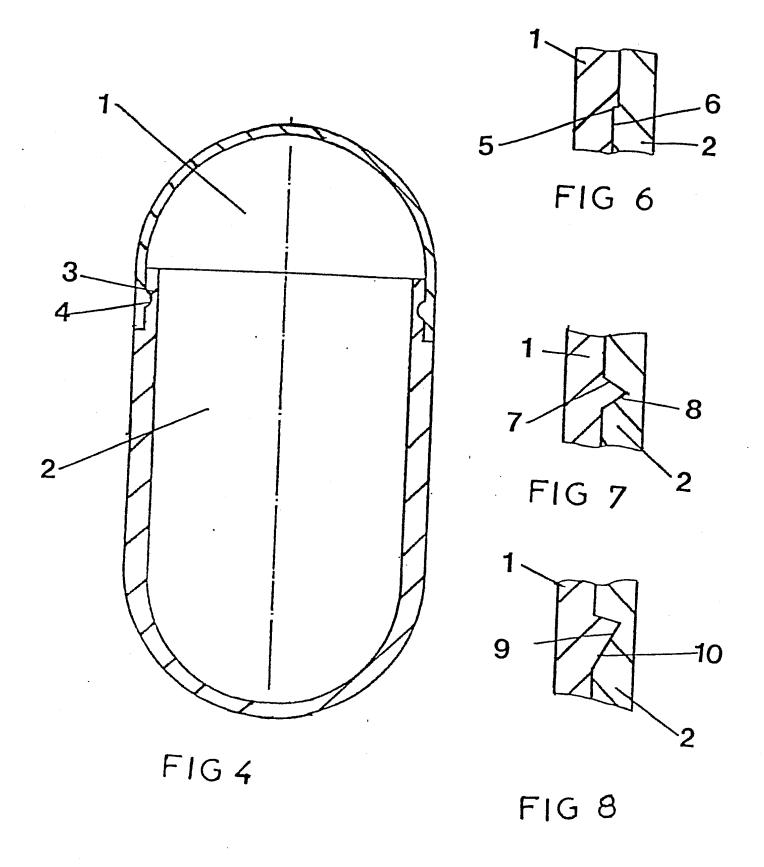


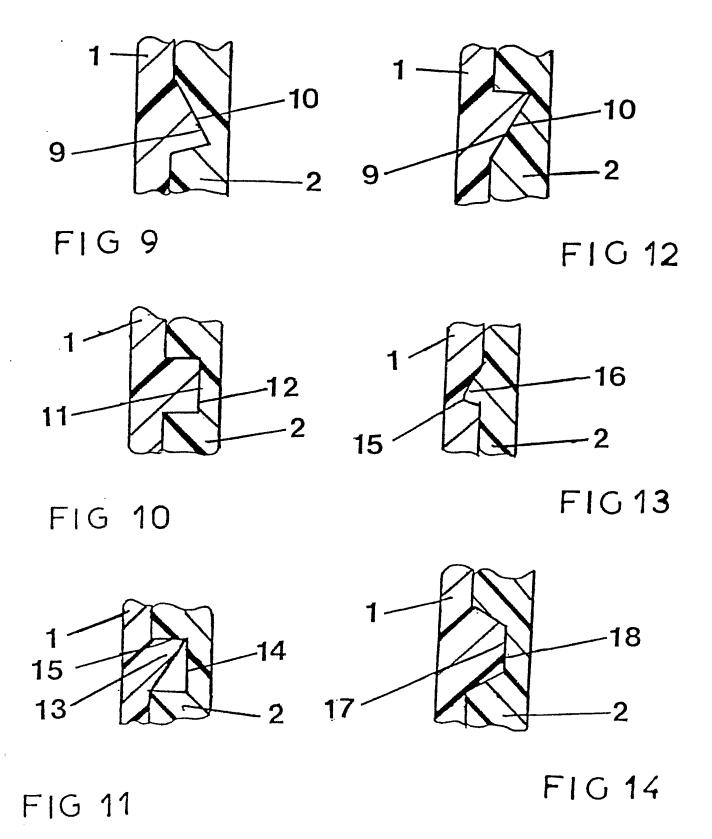
FIG 2



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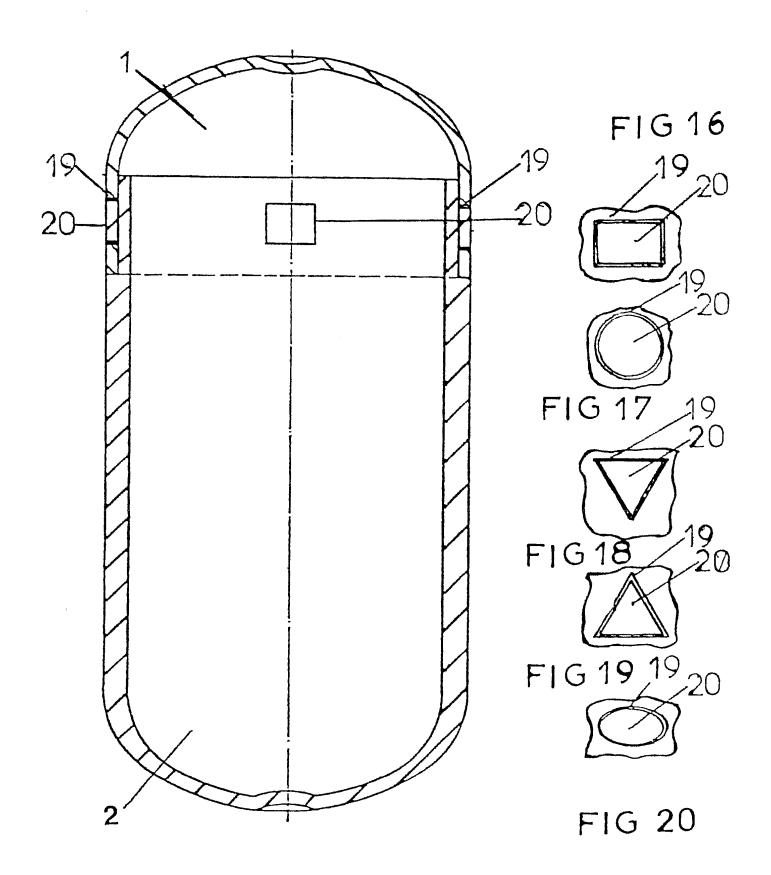


FIG 15

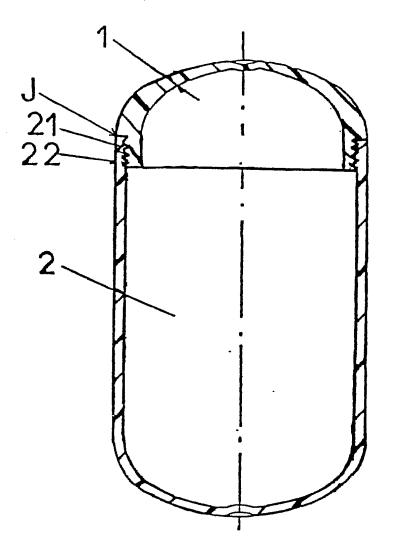


FIG 21

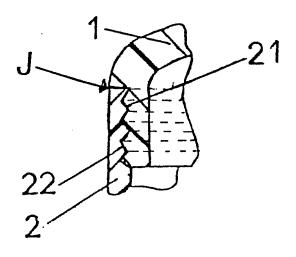


FIG 22

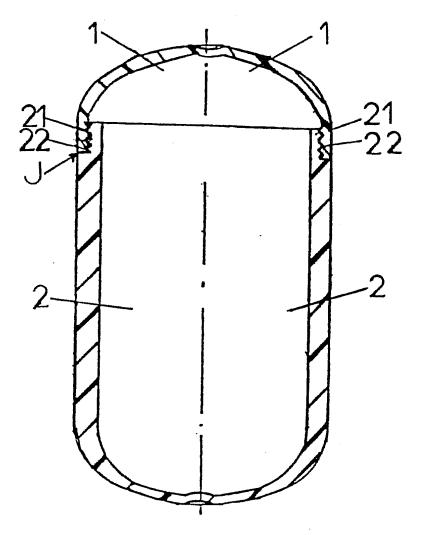


FIG 23

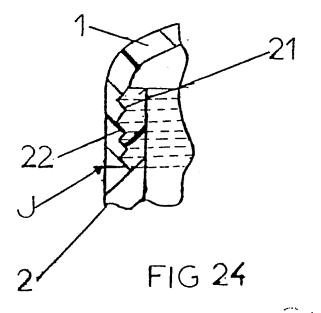
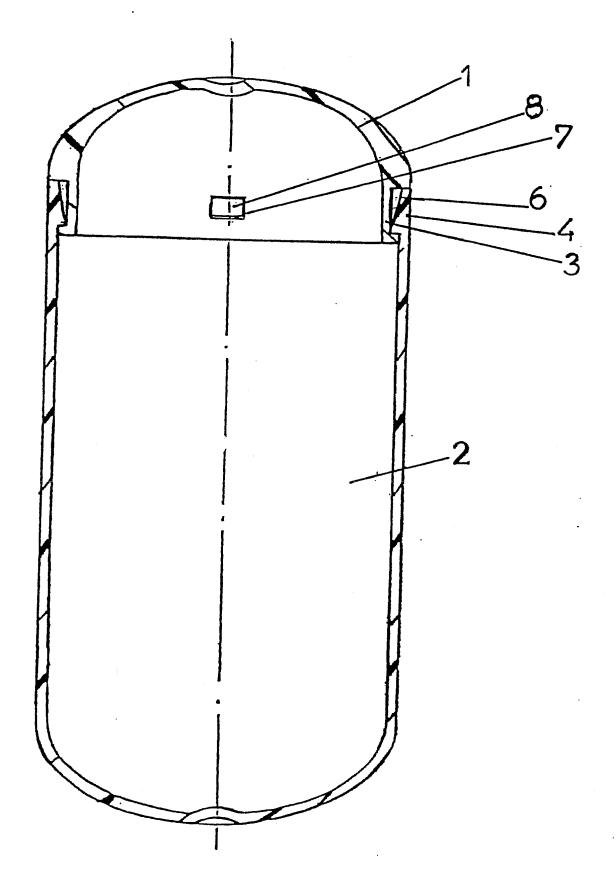


FIG 25



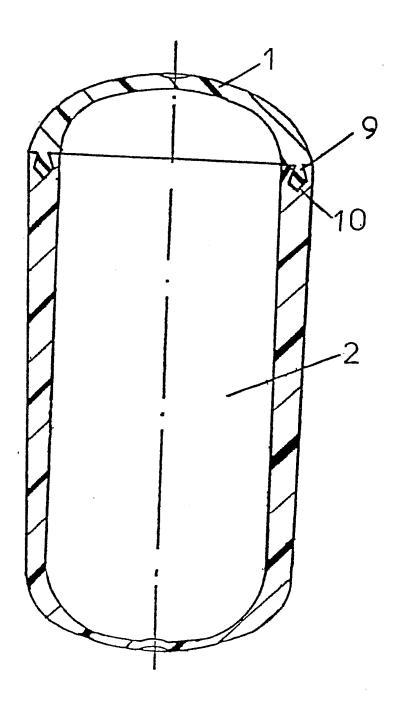


FIG 26

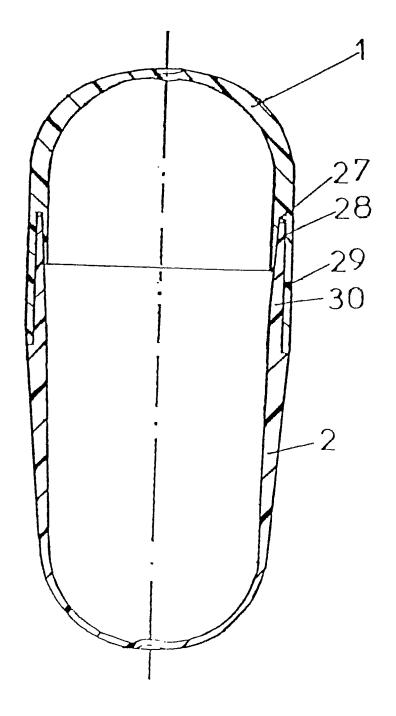
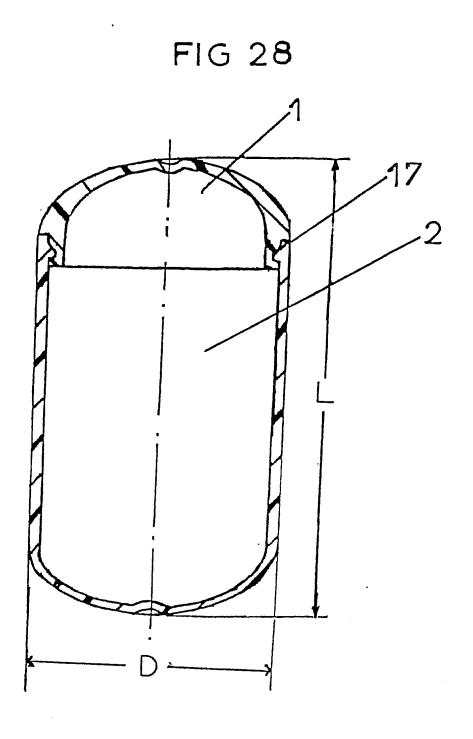
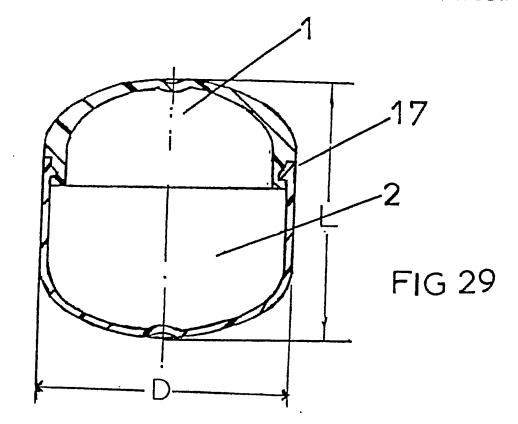


FIG 27





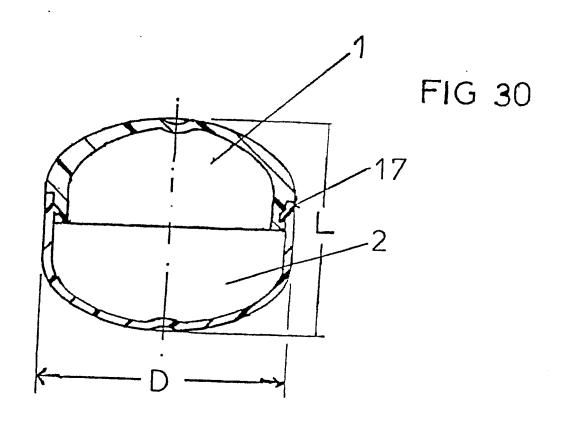


FIG 31

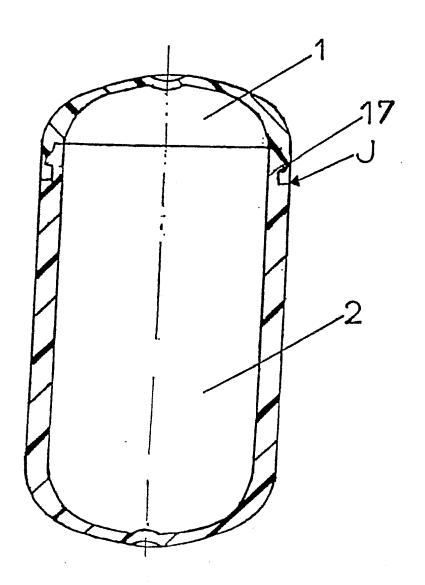


FIG 32

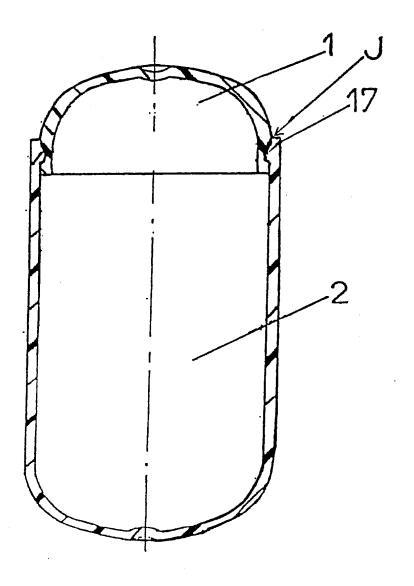
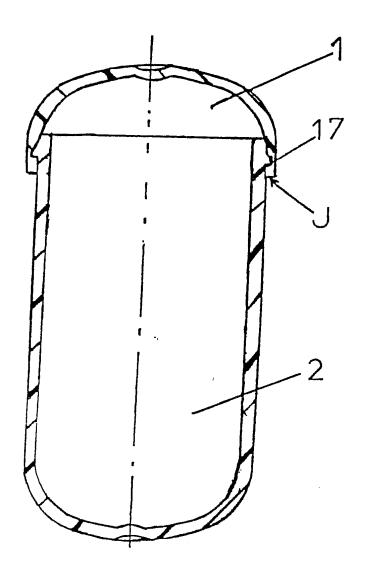


FIG 33



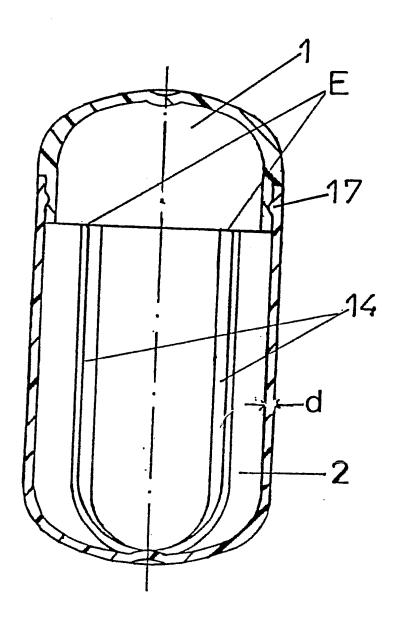


FIG 34

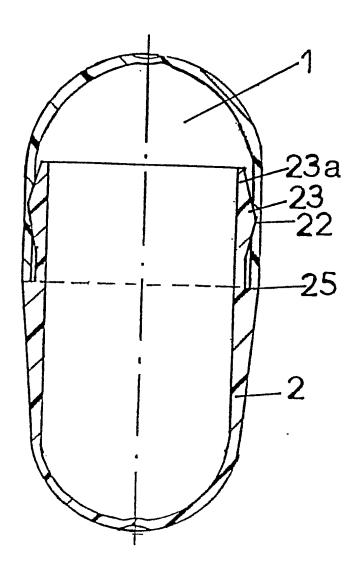


FIG 35

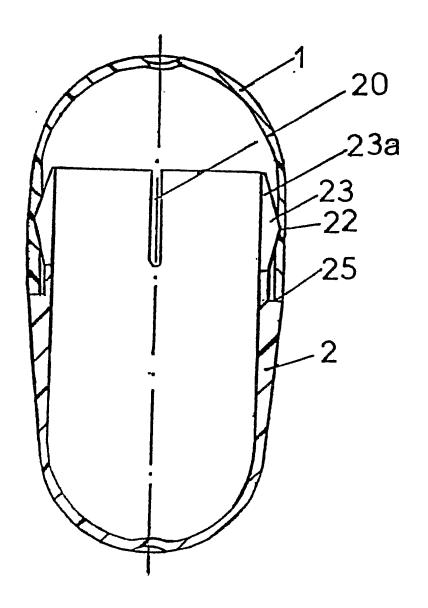


FIG 36

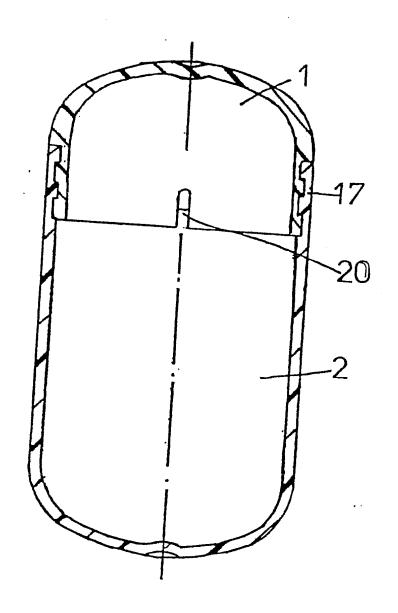
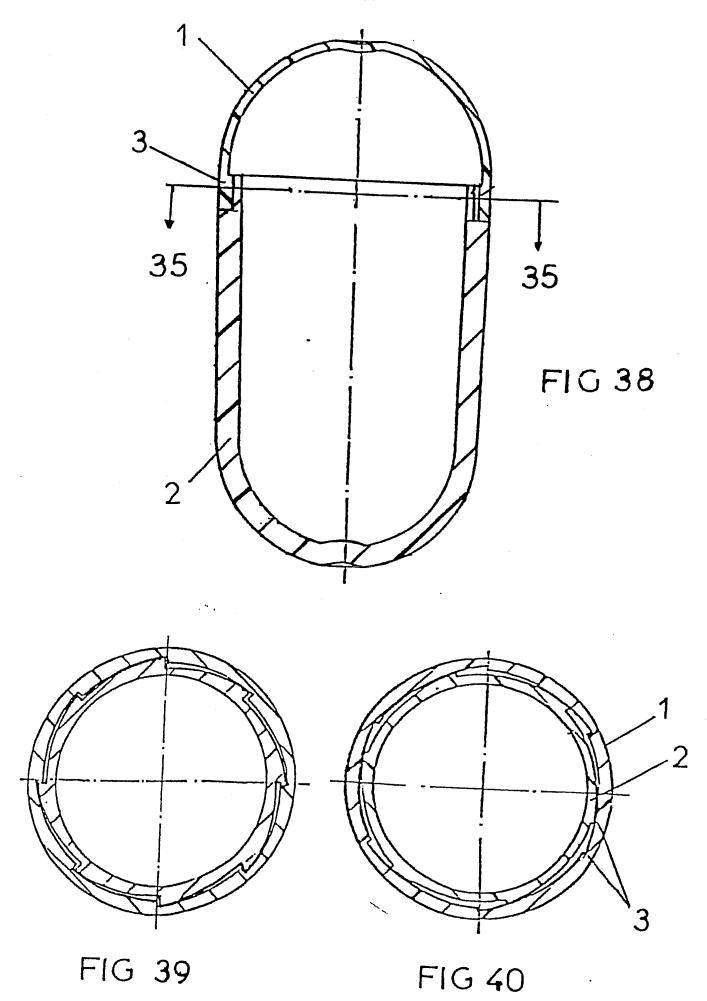


FIG 37



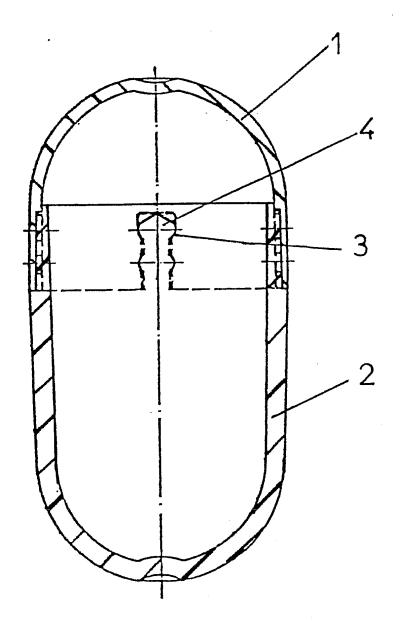


FIG 41

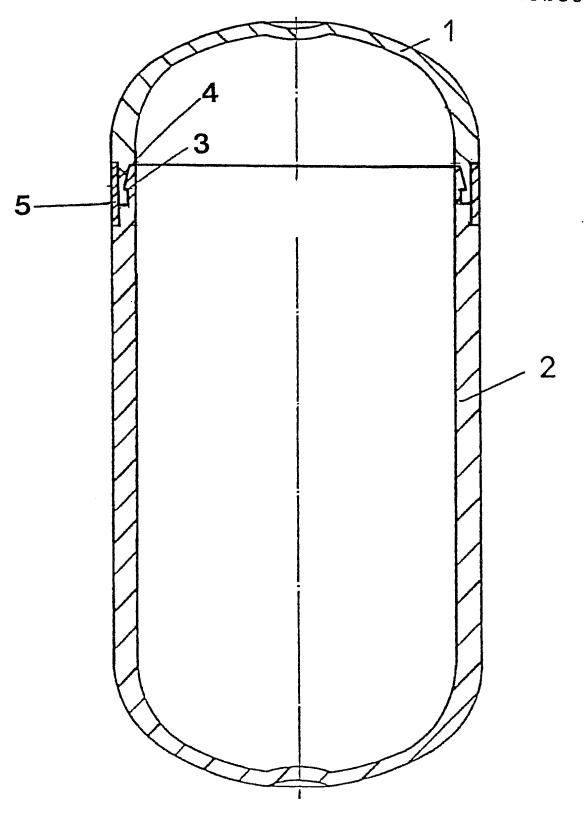


FIG 42

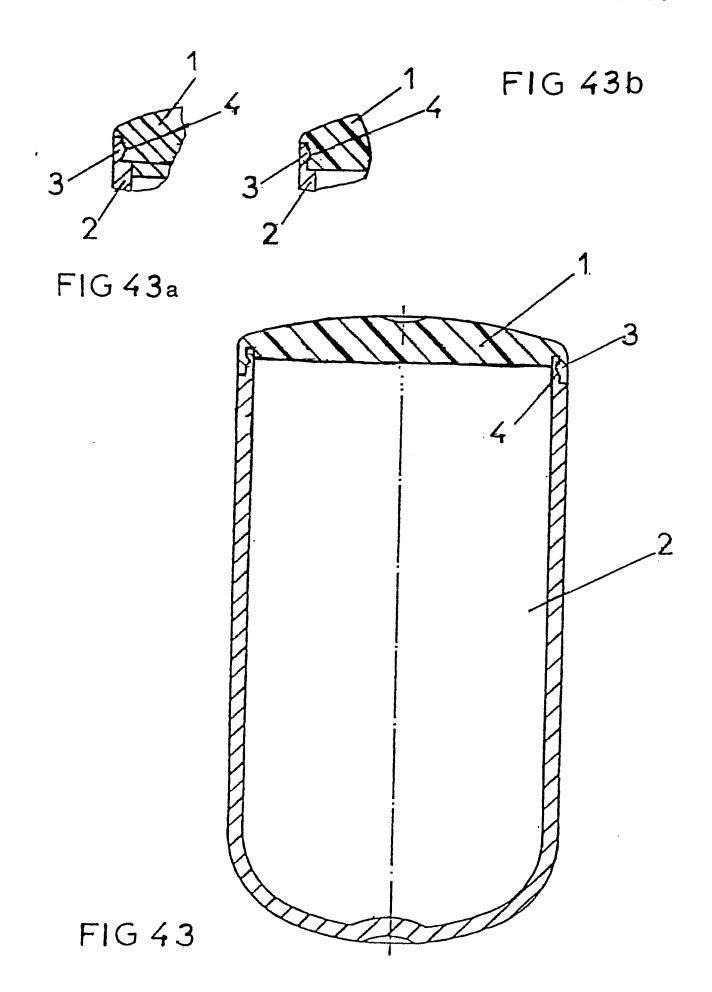


FIG 44

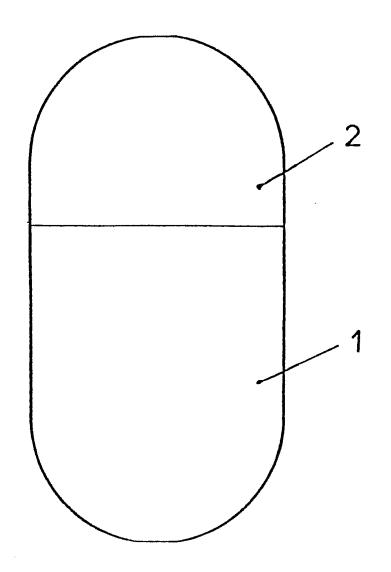
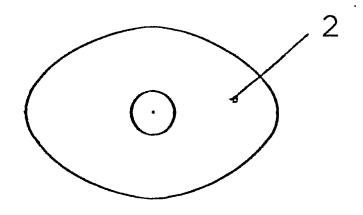


FIG 45



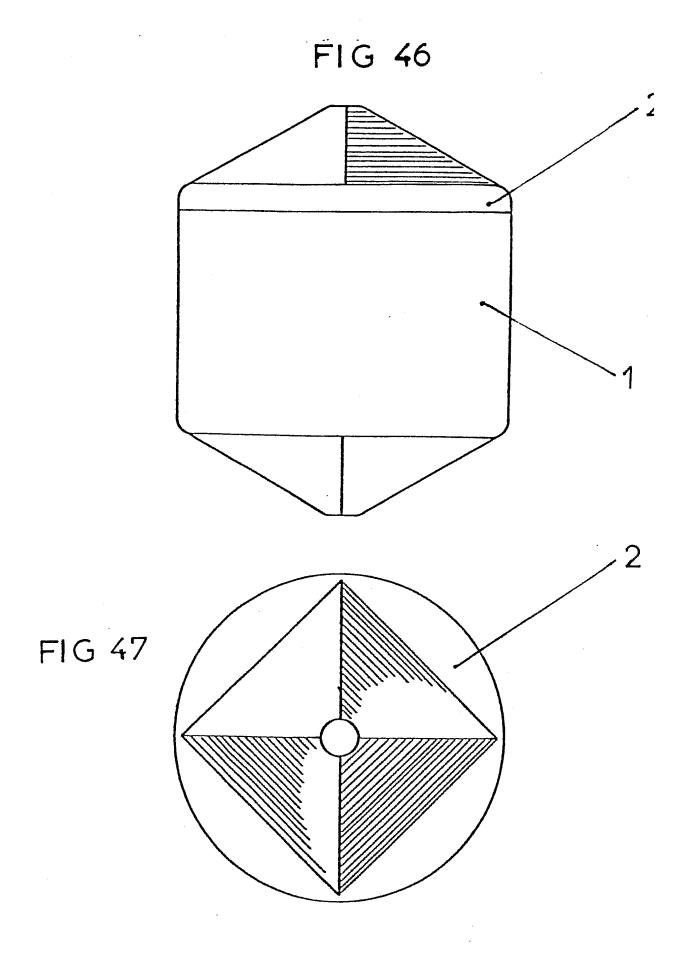
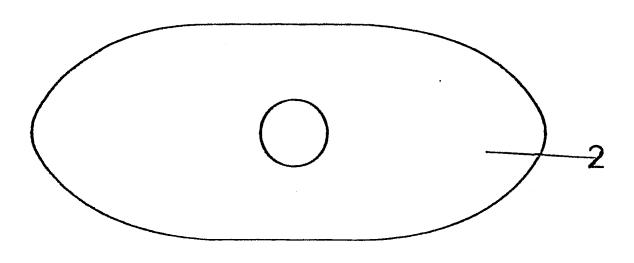


FIG 48



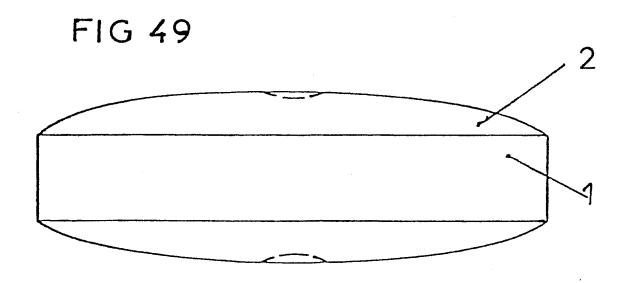


FIG 50

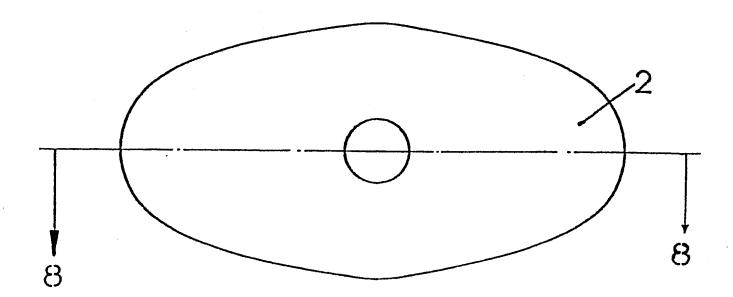


FIG 51

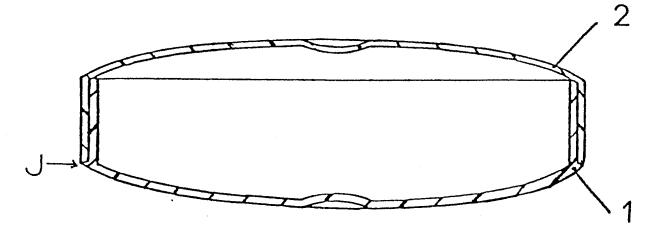


FIG 52

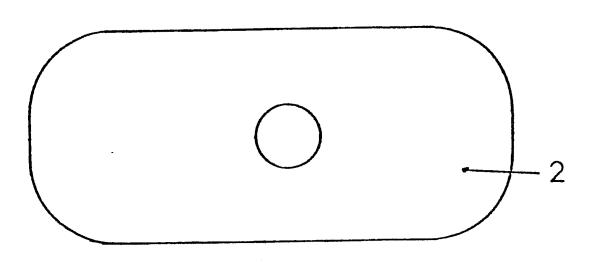


FIG 53

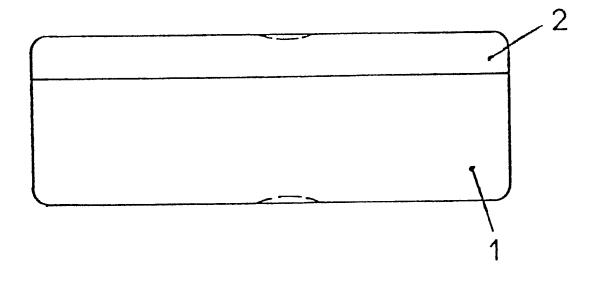
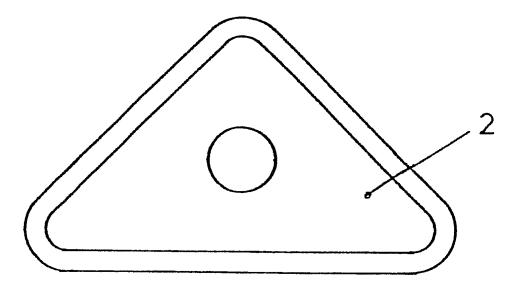
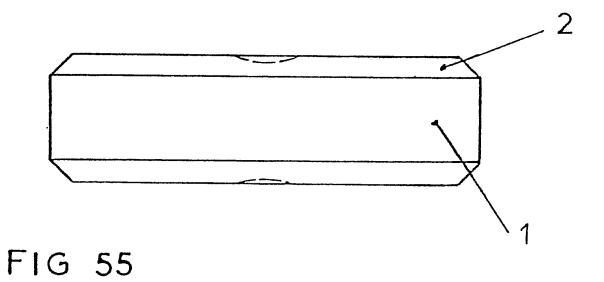
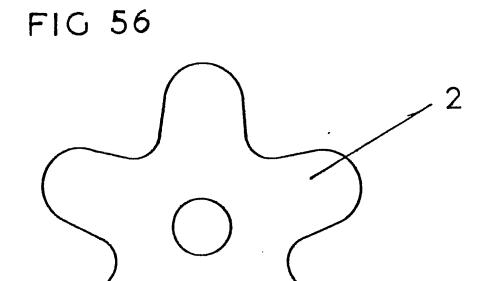


FIG 54







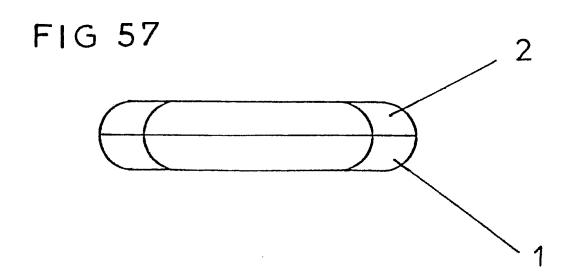
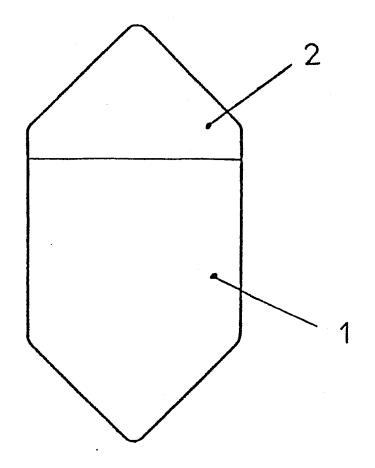


FIG 59



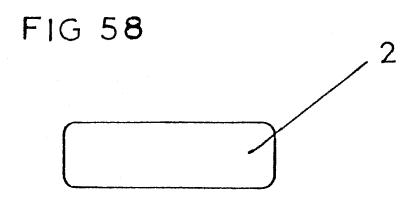


FIG 60

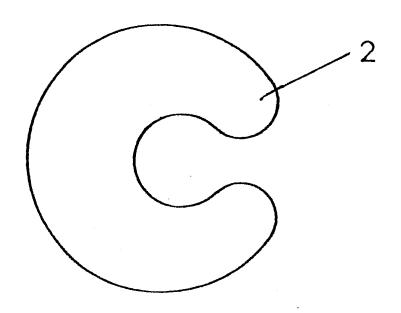


FIG 61

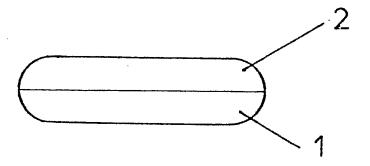


FIG 62

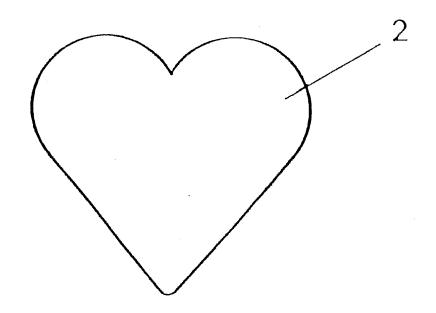


FIG 63 2

FIG 64

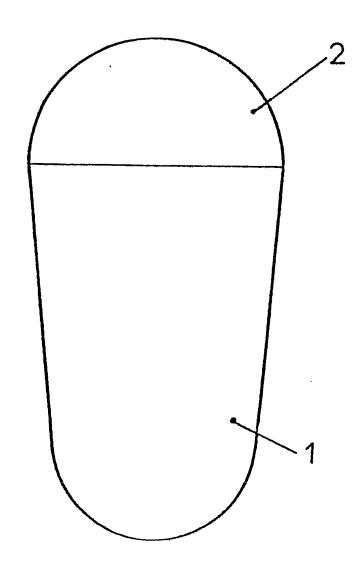
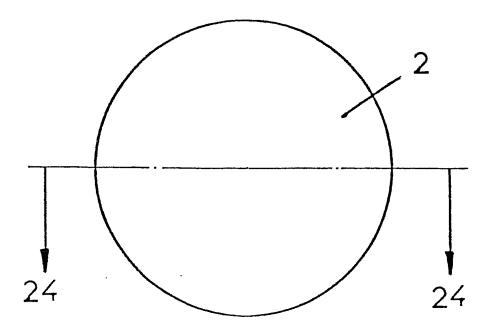


FIG 65

FIG 66



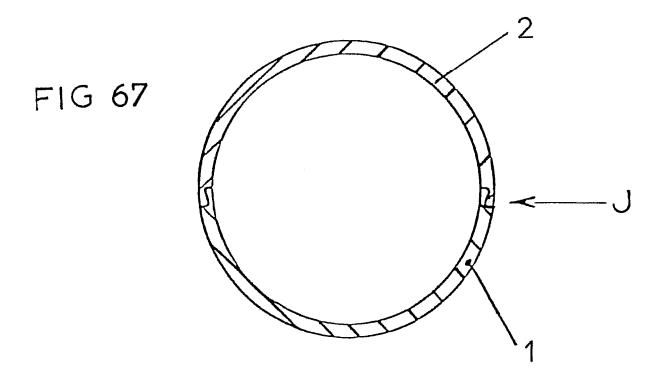


FIG 68

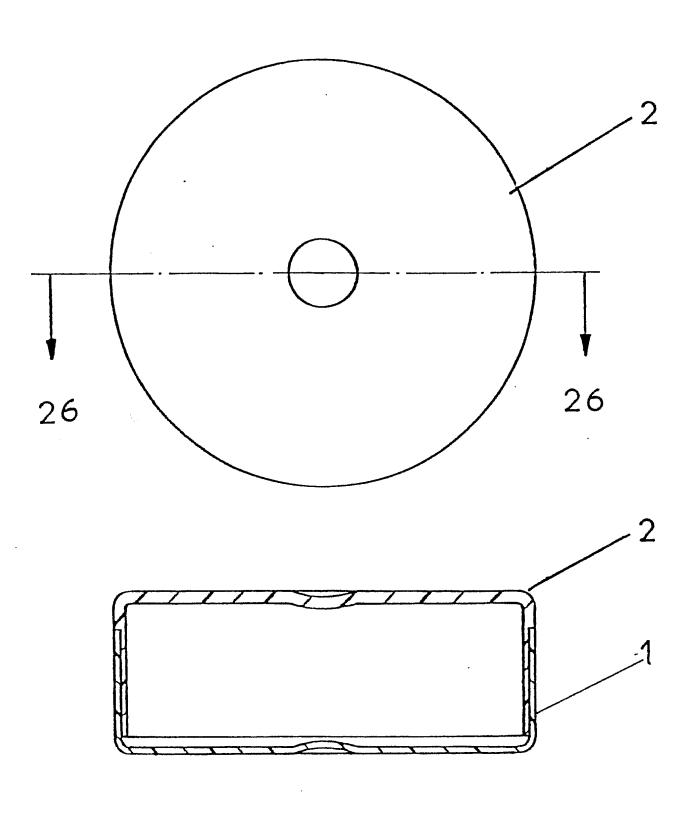
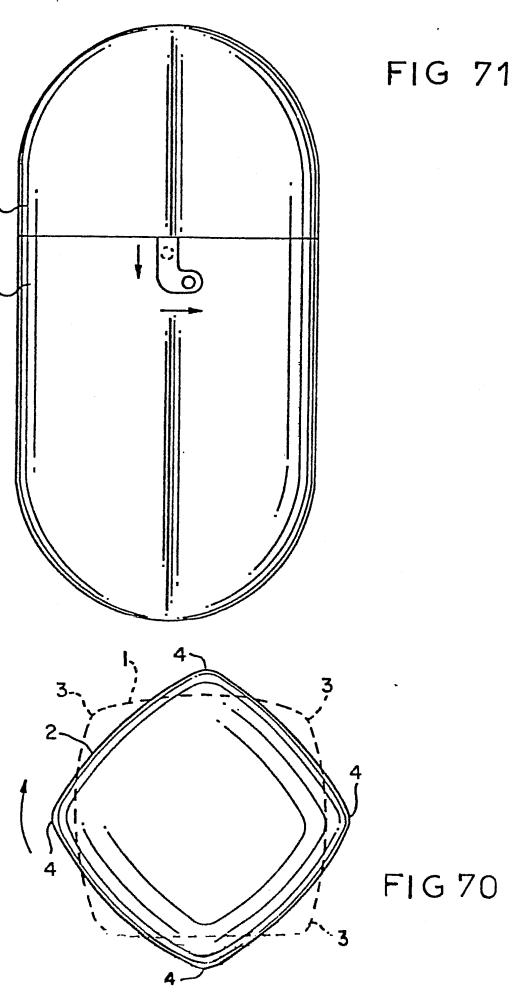
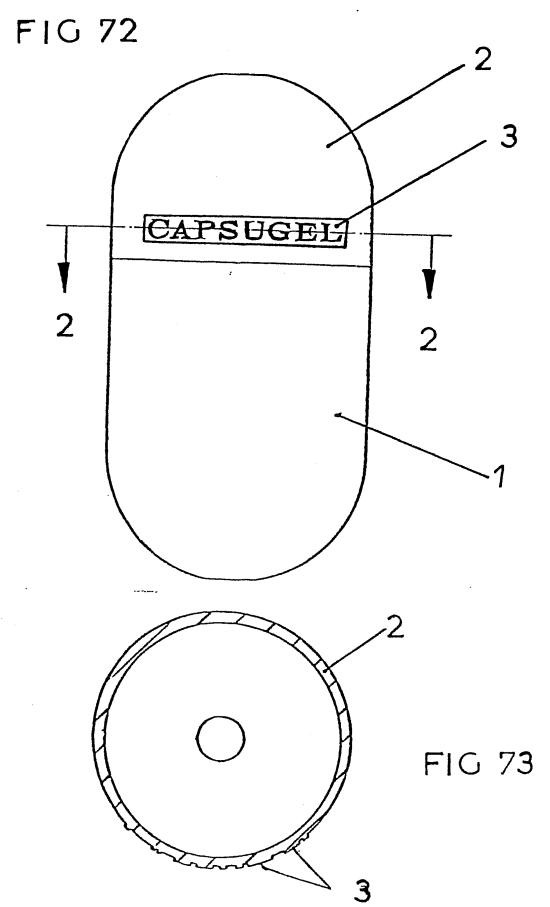


FIG 69





SPECIFICATION

Die-pressure moulded articles

5 The present invention relates to die pressure molded shaped articles formed by die pressure molding of starch, hydrophilic polymers like gelatin or starch derivatives, all with a low water content.

The present invention especially relates to certain structures and forms of such die-molded articles, especially injection molded articles and especially capsules as described hereinafter.

In highspeed die pressure molding such as injection molding it is important that the molding process
15 is carried out at high speed, that the material to be molded shows the quality of a thermoplastic material during the molding process and that the material hardens quickly in the mold so that it can be ejected rapidly without changing its dimensions. The latter
20 is an important factor as the accuracy of very many die pressure molded articles must be within very narrow limits.

It has now been found that these mentioned materials can be die-molded, especially injection-25 molded, to special structures and forms of high accuracy and dimension stability.

The present invention concerns die pressure molded articles and especially injection molded capsules, said capsules having a body part and a cap 30 part, each having a side wall, an open end and a closed end, the two parts being joinable, characterized in that this capsule is made.

 (i) from a hydrophilic polymer or a mixture of such polymers having a water content of 5 - 25% by
 35 weight (calculated to the hyrophilic polymer composition) and if formed from gelatin said gelatin having a water content of 10 - 20% by weight (calculated to the gelatin composition) and/or

(ii) from a starch or a starch composition having a40 water content of 5 - 25% by weight (calculated to the starch composition)

(iii) by die pressure molding and

(iv) that the cap part is die-molded as a stopper directly onto the open end of the body part after the45 body has been filled so as to seal the contents within the capsule, or

(v) that each of said cap and body parts has in the side wall area, adjacent to its open end, at least one locking means, said locking means being arranged 50 to face each other and to achieve, after joining of said parts, a separation-resistant connection, the cap and body parts being worked at a deviation of less than 1% and being stable in dimension.

A special feature (vi) of such articles according to 55 (v) is that at least one part of the open end has the side wall portion recessed with an annular shoulder for receiving and for constituting a stop means in the joining area for the side wall portion at its open end of the other part, both side wall portions being 60 shaped so that the outside surface is smooth in the

A further feature (vii) of such articles according to (v) is that the inner surface of the cap, when said cap is mounted on the open end of said body is at

joining area of said two parts.

horizontal plane touching the open end of the cylindrical side wall of said body, whereby, after the filling of the contents and after the closing of said cap and body, practically no air is entrapped be70 tween said inner surfaces of said cap and the level of said contents.

Articles prepared by the present invention have the further advantage (viii) that they can be diemolded so as to be joined in a distinctive shape to 75 provide a palpable identification of the capsule.

It is also possible (ix) to prepare articles according to the present invention where the cap and/or the body have embossed printing of letters thereon.

A further embodiment (x) of this invention is the complete liquid proof sealing of the cap and body parts by wetting the joining surfaces with water.

The preferred articles are two-piece capsules which have hitherto been made from gelatin by a dip-molding process. Such capsules can, according to this invention, also be made from starch by the method of die-pressure molding. They can be useful for the exact dosage of solid, creamy and liquid substances, especially for pharmaceutical use, said capsule having two parts, a cap part and a body part which are joinable; each of said capsule parts having a side wall, an open end and a closed end.

Preferred articles respectively capsules, according to this invention, are made from starch and by injection molding.

95 The injection molding of hyrophilic materials such as gelatin containing water has been described in the European Patent Application Publication No. 92908.

Hydrophilic polymers are polymers with molecu-100 lar masses from approximately 103 to 107 Dalton carrying molecular groups in their backbone and/or in their side chains and capable of forming and/or participating in hydrogen bridges. Such hydrophilic polymers exhibit in their water adsorption isotherm 105 (in the temperature range between approximately 0 to 200°C) and inflection point close to the water activity point at 0.5. Hydrophilic polymers are distinguished from the group called hydrocolloids by their molecular dispersity. For the maintenance of 110 the molecular dispersity of said hydrophilic polymers a fraction of water - according to the working range of the present invention - of 5 to 25% by weight of said hydrophilic polymers must be included provided that the temperature of said hyd-115 rophilic polymers is in the working range between 50°C and 190°C of the present invention.

Gelatin as a preferred hydrophilic polymer is made from various types of gelatin, including acid or alkaline processed ossein, acid processed pigskin, or 120 alkaline processed cattle hide. Said types of gelatin have a molecular proferably in the mass range of 10.000 to 2 × 10⁷ Dalton or a molecular mass range of 10.000 to 2 × 10⁶ and 10 × 10⁶ to 20 × 10⁶ Dalton. It has a water content of preferably 10 - 19% and 125 especially 12 - 18% by weight calculated to the gelatin composition.

Whilst the die pressure molded articles according to this invention are preferably made from gelatin of various types, all the hydrophilic polymers, gelatin

agar.

to the composition.

plasticizers, lubricants, cross-linking and coloring agents as described in the European Patent Application Publication No. 92908 in concentrations indicated in this European Patent Application. The 5 polymers may also contain extenders up to 95% and preferably not more than 30% by weight such as proteins like sunflower proteins, soybean proteins, cotton seed proteins, peanut proteins, rape seed proteins, lactose, gum arabic, acrylates and methac-10 rylates, water soluble derivatives of cellulose like cellulose acetyl phthalate (CAP), hydroxypropyl cellulose, hydroxypropyl methyl-cellulose, hyroxypropyl methylcellulosephthalate (HPMCP), hydroxymethylcellulose, polyvinylpyrrolidone, shellac, ben-15 tonite, polyvinyl-acetatephthalate, phthalated gelatin, succinated gelatin, polysaccharides like agar-

Some of these extenders as well as others are hydrophilic polymers themselves and can be used to 20 produce articles according to this invention.

Such hydrophilic polymers are hydroxypropyl methylcellulosephthalate (HPMCP), polyvinylacetatephathalate (PVAP), celluloseacetylphthalate (CAP), acrylates and methacrylates (Eudragit), phthalated gelatin, succinated gelatin, crotonic acid, and shellac. These polymers are preferred when enteric properties are required. They contain preferably 5 - 25% of water, preferably 15 - 22% by weight, calculated to the waterfree polymer. Preferred is HPMCP, PVAP and CAP, and specially PVAP and CAP; most preferred is PVAP, all being worked by injection molding having a water content of 5% to 25%, preferably of 15% to 22% by weight, calculated

35 Many of these polymers are non-thermoplastic and it is very surprising that they can be die-pressure molded according to this invention.

Molding temperatures range from 50 to 190° C and pressures from 600 to 3.000×10^{5} N/m² known 40 apparatus can be used.

Preferred is a moldable starch composition made from wheat, potatoes, rice, tapioca and the like.

The starch contains about 0 to 100% of amylose, and about 100 to 0% of amylo-pectin; preferably 0 to 45 70% of amylose, and about 100 to 30% of amylo-pectin and most preferably potato starch and maize starch.

When in the following description the term 'starch' is used, this also includes foams, modifications or 50 derivatives of starch, and combinations thereof with hydrophilic polymer compositions whose properties are acceptable for the intended die-pressure molded products, especially capsule materials.

The starch has a water content of 5 - 25%, 55 preferably of 15 - 22% and especially of 17 - 20% (calculated to the starch composition).

The starch may contain various additives such as plasticizers, lubricants, cross-linking and/or coloring agents, as well as extenders. All these are the same 60 as mentioned above for the hydrophilic materials.

Molding temperatures for starch are 80 - 240°C and pressures ranging from 600 to 3.000×10^5 N/m²,

The starch containing the water and optionally further additives is molten under these temperatures

molding apparatuses known per se.

The skilled in the art will be able to work with die pressure molding, i.e. profile extrusion, crompression molding, vacuum forming, thermal forming, extrusion molding, polymer casting in combination with vacuum forming. Preferred however is injection molding.

According to point (iv), the cap part is diemolded as a stopper directly onto the open end of the body.

75 This works unexpectedly well with the mentioned compositions and the high speed process, preferably injection molding and extrusion, to produce liquid proof capsules. The body may be produced by any known method from a material specified

80 hereinabove.

Detailed description of embodiment (iv):

Figure 1 is a longitudinal sectional view of an embodiment of the capsule of the present invention;

Figure 2 is a longitudinal sectional view of another embodiment; and

Figure 3 is a longitudinal sectional view of a

further embodiment.

In Figure 1 there is shown an embodiment of a capsule which might be filled with a pharmaceutical product to be swallowed by the patient. In this

embodiment the cap-stopper 1 is formed by diemolding so as to provide a smooth outer surface when joined, i.e. it has the same outer diameter as the capsule body 2 which is filled with the contents 3.

Figure 2 shows another embodiment wherein a body 2 is provided with a groove 4 to assure additional tightness of the stopper 1 when joined over the contents 3.

In Figure 3 there is shown a further embodiment
wherein a flat, circular plate or disc 4 was inserted
into the body 2, so as to completely cover the filling
material 3. In this embodiment the stopper 1 is
formed by die-molding so as to provide a smooth
outer surface when joined, i.e. it has the same outer
diameter as the capsule body 2 which is filled with
the material contents 3. The cover plate 4 will permit
injection molding of the closing stopper at a very
high speed.

The present invention provides also the possibility 110 to mold high precision locking articles especially capsules which are liquid- and tamper-proof.

In this application a 'locking capsule' is defined to include a filled and joined capsule wherein the capsule parts are formed so as to impede separation and tampering with the filled contents.

Due to the limitations of manufacture of prior art capsules by the prior art dip-molding process, prior art capsules have a disadvantage that they do not have secure locking means to prevent separation

120 after filling and joining. There is a problem if such a capsule, especially one containing food or drugs, can be opened or tampered with.

It is therefore an object of the present invention to provide a capsule which has a structural configura125 tion so as to prevent tampering. The advantages of the present invention will become apparent to those skilled in the art from a consideration of the detailed description which follows with reference to the accompanying drawings.

Detailed description

Figure 4 is a longitudinal section of a first embodinent.

Figures 6 to 14 are partial sections showing nine 5 further embodiments of Figure 1.

Figure 15 is a longitudinal section of another embodiment.

Figures 16 to 20 are partial sections showing five further embodiments of Figure 15.

10 Figure 22 is a partial sectional enlargement of Figure 21.

Figure 23 is a longitudinal section of another embodiment.

Figure 24 is a partial sectional enlargement of 15 Figure 23.

Figure 25 is a longitudinal section of another embodiment.

Figure 26 is a longitudinal section of another embodiment.

20 Figure 27 is a longitudinal section of another embodiment.

Figures 28 to 30 are longitudinal sections of another embodiment.

Figures 31 to 37 are longitudinal sections of 25 further embodiments.

Figure 38 is a longitudinal section of another embodiment.

Figure 39 is a section along line 35 - 35 of Figure 38.

Figure 40 is a view of Figure 39 after one of its parts has been axially rotated with respect to its other capsule part.

Figure 41 is a side view of a further embodiment.
Figure 42 is a longitudinal section of another
35 embodiment.

The capsule shown in Figure 4 of the drawings has a cap part 1 and a body part 2. The cap part 1 has an annular ridge 3 protruding from the inner surface of the side wall adjacent to the open end of the body

40 part 2 which mates with a recessed annular groove 4 of the body part 2. The ridge 3 and the groove 4 are structurally and dimensionally adapted to each other so that the ridge 3 and the groove 4 are interlocked by snap-in action when the capsule parts are joined.

It is to be understood that:

 - the ridge 3 may be a continuous ring or be constituted by a number of segments or cams cooperating with a continuous or discontinuous groove;

50 - the locking means may comprise one or more ridge and groove structures.

 - the cross-sectional shape of the locking means may comprise not only semicircle forms but any other suitable form such as a triangle, a semi-oval or
 55 other fractions of circles or ovals.

Figures 6 to 14 are alternate embodiments of the capsule shown in Figure 4. In Figure 6 the cap part 1 has a right angle groove 5 which mates with a right angle ridge 6 of cap part 2.

Figure 7 shows another embodiment wherein the cap part 1 has an annular conical ridge 7 which mates and locks with an annular conical groove 8 on body part 1.

Figure 8 shows another embodiment wherein the

mates and locks with a corresponding annular triangular groove 10 on body part 2. For better locking the short side of the triangular ridge 9 faces the short side of the triangular groove 10. It has been 70 found that the optimum locking force occurs when the short side of the triangular ridge 9 faces the open end of the other part. The joining of the capsule parts if facilitated when the shortest side of the triangular ridge facing the open end of the other capsule part 75 forms with the adjacent side wall an angle of about 90 to 134 degrees.

Figure 9 shows another cap part 1 having a triangular ridge 9 in mating and locking engagement with a triangular groove 10 on body part 2.

80 Figure 10 shows a cap part 1 having annular rectangular ring 11 in mating and locking engagement with a corresponding annular groove 12 on body part 2.

Figure 11 shows a cap part 1 having a annular 85 ridge with a triangular cross section 13 wherein the shortest face of the triangle 15 mates and locks with the top surface of a rectangular groove 14 in body part 2.

Figure 12 shows another embodiment of a cap part 1 having a triangular ridge 9 in mating configuration with a triangular groove 10 on body part 2.

Figure 13 shows a cap part 1 having a ridge 15 with the cross sectional area of a bead mating and locking with a corresponding annular groove 16 having a 95 corresponding cross sectional area of a circular segment 1 on body part 2.

Figure 14 is another embodiment showing a cap part 1 having an annular ridge 17 with a generally parallel-epipedonal cross sectional area which mates and locks with an annular groove 18 having a corresponding parallel-epipedonal cross sectional area on body part 2.

Figure 15 another embodiment of the invention showing a cap part 1 having one or more windows 105 19 arranged on its cylindrical side walls near the open end and on a circular path which is coaxial with the axis of the capsule. The body part 2 has a corresponding number of locking cams 20 which are protuberances on its cylindrical side walls near its 110 open end. Said cams 20 mate with the windows 19 in locking engagement when the capsule parts are ioined.

Figures 16 to 20 show alternate embodiments of the locking window 19 and cam 20 of Figure 15; in 115 Figure 16, the cross sectional area is rectangular; in Figure 17, circular; in Figures 18 and 19, triangular; in Figure 20, oval.

Figure 21 shows an alternate embodiment of the present invention wherein the cap part 1 has a male thread 21 on the outside surface of its cylindrical side walls at its open end. The male thread 21 engages with a female thread 22 on the inside surface of the body part 2 at its open end. It is another feature of this embodiment that the body part 1 and the cap part 2 can be joined with an outside smooth surface, as at J, so as to make separation difficult, thereby

enhancing the locking feature of the capsule.
Figure 22 is a partial enlargement of Figure 21
showing the mating engagement of the male thread

surface J at the joining area.

Figure 23 is an alternate embodiment of Figure 21 wherein the body part 2 has a male thread at the outside surface of its open end. The male thread 21 mates and engages with a female thread 22 on the inside surface at the open end of cap part 1.

Figure 24 is a partial enlargement of Figure 23 showing the smooth surface J at the joining area.

Figure 25 shows a further embodiment of the
10 invention having a bayonet-tape locking arrangement wherein the cap part 1 has an annular rectangular groove 3 adjacent to the open end of the cap 1 in a direction generally parallels to the capsule axis.

The body part has a triangular ridge 4 on the inside
15 surface at the open end of the body part 2. The triangular ridge 4 has a conical taper 6 at the leading edge in order that the open end of body part 2 can enter more easily within body part 1. In addition, cap part 1 has a window 7 for mating with a protruding

20 cam 8 on cap part 1. The combination of the groove 3 engagement with ridge 4 plus the window 7 with the cam 8 gives a secure bayonet-tape lock engagement to enhance the locking aspect of this embodiment.

Figure 26 is an alternate embodiment of the
25 invention wherein the cap part 1 has an annular
dove-tail ring 9 on its cylindrical side wall at its open
end for mating engagement with a dove-tailed
groove 10 on the cylindrical side wall at the open end
of the body part 2.

Figure 27 shows an alternate embodiment of the invention wherein the cap 1 is provided on its cylindrical side wall at its open end with an annular slit 27 which is symmetrically arranged with respect to the main axis of the capsule. The slit 27 is defined by two annular wall parts 28, 29 of different length. The body part 2 has an upright side wall 31 of reduced thickness and tapering towards its open end. In the joined position shown in Figure 27, the side wall 31 of the body 2 is held by flexible pressure to in the annular slit 27 which preferably has a tapered configuration.

According to Figures 28, 29 and 30, a capsule is shown having a cap 1 joined with a body 2. At the joining area a locking means, as at 17, is provided in 45 accordance with the embodiments previously disclosed. It is a feature of the present invention that the ratio can be variable between the outside diameter of the cylindrical side walls (D) and the overall length of the joined capsule (L). Figure 28 shows the

50 capsule embodiment wherein D is less than L. In Figure 29, the capsule embodiment has a ratio of D equal to L. According to Figure 30, the ratio of D is greater than L. The advantages of a variable D to L ratio are that:

 -The volumetric contents of the capsule can be changed to meet particular requirements, especially for pharmaceutical and food use; and

- the configuration can be varied to enable easier swallowing of the capsule, especially for phar60 maceutical and food use, with children, adults and geriatric patients who differ markedly in their ability to swallow capsule medicaments. So a tablet shaped form can be obtained.

Figures 31, 32 and 33 show different embodiments of the invention basing a san part 1 and a back part 3

with a locking means, as at 17, in accordance with the embodiments previously disclosed. Figure 31 shows a feature of the present invention that the locking means 17 can be utilized with a capsule 70 having a smooth outside surface at the joinder J.

Figure 32 shows a capsule having a body part 2 with a protruding edge at the joinder J.

Figure 33 shows a capsule having a cap part 1 with a protruding edge at J.

75 Figure 34 shows a further embodiment of the present invention. The cap part 1 and the body part 2 have a locking means, a at 17, as previously disclosed. In addition, the body 2 has on its side wall inner surface a number of reinforcing ribs 14,

0 molded with protruding from the inner side wall surface. The ribs preferably extend over the whole length of the body part 2 and join each other in the center of the closed end of the body 2. In the construction of said ribs 14, which may be of

85 triangular, rectangular or any other cross section, the bending strength or rigidity of the body 2 is increased to such an extent that the wall thickness D may be substantially reduced. In addition, the upper end faces E of the ribs 14 form a stop and support

90 means for the cap 1. The body 2 may be manufactured by injection molding, and the ribs 14 constitute a flow path for the injected materials so that the quick and regular distribution of said material is facilitated within the injection mold.

Alternatively, the ribs in the above figure could be undercut into the side of one or both parts so as to improve the disintegration of the capsule in the gastro-intestinal tract of the patient. Also, the ribs 14 could be molded with and protruded from the side
 wall outer surface of the body part 2.

In the capsule embodiment of Figure 35, a cap 1 has on the inner surface of its cylindrical side wall an annular groove 22 receiving a conically shaped portion 23 of the cylindrical side wall of the body 2.

The open end of the cap 1 rests upon the annular

The open end of the cap 1 rests upon the annular surface 25 of a shoulder formed in the side wall of the body 2. Joining of the cap and body is facilitated by tapered closed end 23a of body part 2.

In Figure 36 the reference numerals of Figure 35
110 are used for all parts which have remained unchanged. The cap 1 is identical to that shown in Figure 35. However, the body part 2 is additionally provided with a slit 20 - (one or more circumferentially arranged slits may be provided) - which confers upon the open end of the body a greater flexibility thereby assisting and simplifying the joining of cap and body.

Figure 37 shows an alternate embodiment of the present invention wherein the cap parts 1 and 2 are provided with two or more locking means as at 17. In addition, the cap part is provided with a slit means 20 for greater flexibility which assists and simplifies the joining operation of cap 1 and body 2.

Figure 38 shows an alternative embodiment of the 125 present invention wherein the cap part 1 and the body part 2 are each provided with one or more ratchet teeth 3 on their surfaces facing each other.

Figure 39 is a sectional view of Figure 38 showing the teeth 3 when in mating engagement with each

Figure 40 is a sectional view of Figure 38 showing the teeth when not in mating engagement with each other. The application of torque in coaxial rotations of one of the capsule parts around the other capsule part causes a frictional and locking engagement of the teeth of one capsule part upon the facing surface of the other capsule part.

Figure 41 is a side view of a further embodiment of the present invention wherein the body part 2 is 10 provided with one or more protrusions 4 on the outside surface of its side wall adjacent the open end which snaps into locking engagement with a corresponding recess 3 in the side wall of the cap part 1.

Figure 42 is a still further embodiment of the 15 present invention showing the cap part 1 and the body part 2 in a locking engagement 3; both the cap part 1 and the body part 2 have a recessed groove 4 in the joining area which is filled by a band 5 of sealing or bonding material.

In any of the above embodiments, the body may have a reduced diameter at the open end to facilitate entry during joining of the capsule parts.

Figure 4 to 31, 34-42 also show the embodiment as defined above under (vi) where the outside surface 25 of the molded article, especially of the injection molded capsule, is smooth in the joining area of the cap and the body.

The present invention also provides articles, especially capsules, where the open end is closed by a coaxial cap characterized in that the inner surface of the cap, when the cap is mounted on the open end of the filled body, is at practically the same level or below the level of the plane touching the open end of the cylindrical side wall of the body, thereby avoiding air entrapment within the contents of the capsule.

Due to the limitations of manufacture of prior art capsules by the prior art dip-molding process, prior art capsules have the following disadvantages:

- the air entrapped in the spherical closed end of the cap part of the filled and closed capsule results in a moving bubble of air when the content is a liquid;
- numerous capsule contents, especially those which are creamy or liquid, deteriorate after expo-45 sure to the oxygen in the entrapped air;
 - capsules are neither liquid- nor gas-tight;
- the capsules are neither tamper-proof nor separation-resistant. There is a disadvantage if such a capsule, especially one containing food or drugs,
 can be opened or tampered with; and
- when used for pharmaceutical purposes, the protruding edge located on the periphery of the open end of the capsule is relatively sharp. The removal of the protruding edge would make the
 55 capsule more attractive to be swallowed.

It is therefore an object of the present invention to provide a capsule which has a structural configuration so as to avoid the afore-mentioned disadvantages. The capsule of the present invention is separation-resistant. Furthermore, air entrapment during filling is avoided.

Figure 43, 43a and 43b show such an embodiment as a longitudinal view. Of course, locking devices as shown in the foregoing figures may as well be used.

to prepare distinctive shapes by die-pressure molding of the above described materials.

Prior art pharmaceutical capsules have axially joinable cylindrical cap and body parts which require 70 that the inner diameter of the cap side wall frictionally engage the outer diameter of the body side wall. When the cap and the body are joined, the open end of the cap forms a relatively sharp protruding edge. The prior art capsules have the following principal 75 disadvantages:

 due to the limitations of manufacture by the prior art dip-molding process, the prior art capsules cannot differ much in their shapes and are, therefore, not very adaptable with regard to their shapes;

 - identification of the capsule contents must be indicated by means of different colors and imprinting, i.e. only by visual means and not by visual means and of palpable characteristics; and

confusion of prior art capsules with different
 contents may occur because of the limited number of distinctive shapes available.

These principal disadvantages are becoming more serious because the number of oral medications is increasing and there are only a limited number of visual means for identification.

Also prior art capsules have the following limitations:

- they cannot be provided with a smooth outer surface in order to achieve an additional easy-to-95 swallow effect; and
 - they have a relatively large empty space which causes a waste of material and of package volume.

It is therefore an object of the present invention to provide a capsule which has an adapted shape so as to avoid the aforementioned disadvantages and limitations.

The advantages of the present invention will become apparent to those skilled in the art from a consideration of the detailed description which follows with reference to the accompanying drawings.

In the detailed description it is convenient to group the embodiments of the present invention into categories:

110 1. Capsules with a shape which is distinctive
The distinctively shaped capsules of this group
help to avoid confusion and to support a palpable
identification of the capsule.

For convenience, all of the figures in this applica-115 tion use the same reference numerals.

Figure 44 is a side view of a distinctively shaped capsule of the present invention having a cap part 1 and a body part 2. Figure 45 is a top view of Figure 43 showing the novel oval-shaped capsule.

120 Figure 46 is a side view of an alternative embodiment. Figure 47 is a top plan view of Figure 46 showing the pyramidical end-shaped capsule.

Figure 48 is a side view of another embodiment showing a flat shaped capsule. Figure 49 is a top plan view of Figure 48 showing the elongated oval-shaped capsule. Figure 50 is a refinement of the embodiment of Figures 48 and 49 showing a modified oval-shaped capsule. Figure 51 is a sectional side view of Figure 50 showing the side walls of cap

part 1 when joined. Also shown is the smooth outer surface of side walls at the joining area J. The use of completely overlapping side walls and a smooth outer surface at the joining make it difficult for 5 potential tamperers to grip and separate the capsule parts. Figure 52 is a top plan view of another refinement of the embodiments of Figures 48, 49, 50 and 51 showing a rectangular oval-shaped capsule. Figure 53 is a side view of the embodiment of Figure 10 52.

Figure 54 is a top plan view of a further embodiment showing a triangular-shaped capsule. Figure 55 is a side view of Figure 54.

Figure 56 is a top plan view of a still further 15 embodiment showing a star-shaped capsule. Figure 57 is a side view of Figure 56.

2. Capsules with a shape which corresponds to a vendor's logogram.

The logogram-shaped capsules of this group help 20 to identify the vendor. The logogram of imaginary companies have been given in Figures 58-61.

Figure 58 is a top plan view of an embodiment showing a parallelolipodonal-shaped capsule logogram. Figure 59 is a side view of Figure 58.

25 Figure 60 is a top plan view of another embodiment showing a C-shaped capsule logogram. With the use of the new die-molding processes to produce capsules, a great variety of shapes of capsules, including all the letters of the alphabet, is not

30 obtainable, in marked contrast to the severe limitations of prior art hard shell capsule shapes made with the prior art dip-molding method.

Figure 61 is a side view of Figure 60.

3. Capsules with a shape which indicates its 35 purpose.

The purpose-shaped capsules of this group help to indicate their field of application. As shown in Figures 62 and 63, the heart shape indicates the field of coronary applications.

40 4. Capsules with a shape for its dosage form The dosage-shaped capsules of this group indicate their use for other than oral medications. Figure 64 and 65 show a suppository-shaped capsule suitable for rectal medication. It will be understood by those 45 skilled in the art that the embodiments above can enable the production of two-piece hard shell capsules with palpable shapes that can be recognized by visually impaired patients.

5. Capsules with a shape which provides a varying 50 volume of contents.

The embodiments of this group provide varying volumes of contents for the same size of capsule. Figure 67 shows a spherical shape providing the smallest possible package for the largest volume of 55 contents.

Figure 67 is a sectional view of Figure 66 along line 24-24, also showing separation-resistant locking means at the side walls of body part 1 and cap part 2 at the joining area J. Figure 68 is a top plan view 60 showing a circular-shaped capsule having the same diameter as the spherical-shaped capsule of Figures 66 and 67. Figure 69 is a side view of Figure 68 showing the disc-shaped configuration which provides one of the largest possible packages for the smallest volume of contents. The great variety of

capsule shapes by the new die-molding process provides a great flexibility in providing hard shell capsules with varying package to volume rations.

6. Capsules with a shape which enables a precise 70 positioning on a joining machine.

There are various capsules which need an exact positioning of the two capsule parts before joining, e.g. a threaded or bayonette-type lock engagement. This precise positioning can be achieved by means

75 of a locator on the outer surface of the capsule. Figure 70 is a side view of an embodiment of this group. Figure 61 is a top plan view of Figure 70 showing 4 locating positions possible with respect to the rotary angle around the capsule axis. This is a big 80 advantage over a conventional dip-molded capsule which has a circular cross-section area. A capsule with four possible positions is shown having a bayonet-type lock engagement in Figure 70 consisting of e.g. four protruding cams 3 on one part which 85 mates with four corresponding ridges 4 on the other part. A bayonettype closure system is shown in

of the body part, with the cap part 2. (Axial and rotational movement is shown by dotted lines). It will be understood by those skilled in the art that there are capsule shapes of the present invention which meet the characteristics of more than one of

Figure 71 having an axial and a rotational movement

the above mentioned groups. In a further embodiment of the invention the 95 die-pressure molded articles are manufactured so as to provide bossed imprinting of letters or designs thereon.

Prior art capsules are imprinted by means of an ink composition. This process is very complicated and, 100 in addition, it requires a second step after the manufacturing of the capsules. Furthermore, it is difficult to imprint the closed ends of the capsule body and cap parts. With the bossed imprinting of the present invention it is possible to obtain an 105 imprinted capsule without the use of chemical inks. Thus, the imprinted capsule may be fully natural. It is therefore an imprinting of letters or designs so a to avoid the aforementioned disadvantages.

The present invention was achieved by means of 110 die-molding, the capsule parts with a mold which is provided with the desired imprinting (debossed or embossed) of letters or designs thereon.

In the present invention, the capsule manufacturing and the imprinting consist of single and simple steps. Furthermore, during debossing by injection molding, the capsule material is not adversely affected as in the prior art when the imprinting is made by a hot stamp. When a hot stamp is used there is also the disadvantage that a second proces-120 sing step is necessary. Also, with a hot stamp, an embossing is not possible.

Figure 72 is a side elevated view of an embodiment of the present invention showing a joined capsule with a body part 1 and a cap part 2.

125 Imprinted on the outside surface of the side wall of the cap is the embossing 3 of the letters in the name of the vendor CAPSUGEL. Such imprint was not possible with the prior art dip-molding process, but embossing can be achieved with the new die-

Figure 72 along line 2-2.

The present invention may also include sealing or bonding of the joined capsule parts provides an additional securing which further impedes separation and tampering. Such sealing or bonding also makes the capsule liquid, moisture vapor and gastight.

For sealing gelatin it is appropriate to wet the joining surfaces of the cap and body parts with

10 water, either with water alone or a mixture of water with a water miscible organic solvent like an alcohol with 1-4 carbon atoms like ethanol. Optionally the capsule is heated to about 40 - 75°C. Due to the high precision and the special locking means achieved by

15 this die-pressure molding, heating is not absolutely necessary through it improves coniderably the sealing quality. For starch and many of the starch derivatives it was surprisingly found that wetting the joining surfaces of the cap and the body part with

20 water at room temperature without additional heating brings an excellent effect, the capsule being absolutely liquid and tamperproof.

It should be understood that this disclosure is for the purpose of illustration only and that the present 25 invention includes all modifications and equivalents falling within the scope of the appended claims.

Example 1

A capsule body was die-molded on an injection 30 molding machine. The material was bone gelatin, 150 Bloom, B grade, having a water content of 17% based on the weight of the composition. The material was plasticized at 130°C and injected into a die at 20°C. The capsule body was then removed from the 35 mold, filled with lactose and inserted into a second mold. The second mold was mounted in the injection molding machine wherein the stopper was injection molded onto the open end of the filled capsule body. The material of the stopper was the 40 same as for the body. During injection molding of the stopper, a seal was formed between the stopper and the body so that the capsule was liquid, moisture vapor and gas-tight as well as tamperproof.

Example 2

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A capsule body was die-molded on an injection molding machine. The material was bone gelatin, 150 Bloom, B grade, having a water content of 10% and a content of organic plasticizers: sorbitol of 6% and glycerol of 3%, all based on the weight of the gelatin. The material was plasticized at 130°C and injected into a die at 19°C.

After removing the capsule body from the mold, it was filled with a creamy content, Labrafil^R, trade-mark owned and material supplied by Gattefosse, 39 ave Ed. - Vaillant 92100 Boulogne/France, for C12 to C13 exthoxylated saturated glycerides. Then the procedure described in Example 1 above was followed.

Example 3

A wheat starch containing 20.6% of water was injection molded to a capsule body at 140°C and a

the same conditions. The cap and body having the form as disclosed in Figure 4. An excellent locking capsule was obtained with smooth outer surface.

70 Example 4

Example 3 was repeated using the form of Figure 8. The joining surfaces of the cap and body parts where wetted with water. Excellent sealing was the result.

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Example 5

Example 3 was repeated using the form of Figure 43, having embossed the name CAPSUGEL on the cap, with very good result with respect to air-80 entrapment and sealing quality. The depth of the embossed letters was 0.03mm which was sufficient for clean reading. The same result is obtained with debossing.

85 CLAIMS

- Die pressure molded articles and especially injection molded capsules, said capsules having a body part and a cap part, each having a side wall, an open end and a close end, the two parts being joinable, characterized in that these articles are made
- (i) from a hydrophilic polymer or a mixture of such polymers having a water content of 5 25% by
 95 weight (calculated to the hydrophilic polymer composition) and if formed from gelatin said gelatin having a water content of 10 20% by weight (calculated to the gelatin composition) and/or
- (ii) from a starch or a starch composition having a100 water content of 5 25% by weight (calculated to the starch composition)
 - (iii) by die pressure molding and -for capsules-
- (iv) that the cap part is die-molded as a stopper directly onto the open end of the body part after the
 body has been filled so as to seal the contents within the capsule, or
- (v) that each of said cap and body part has in the side wall area, adjacent to its open end, at least one locking means, said locking means being arranged
 110 to face each other and to achieve, after joining of said parts, a separation-resistant connection, the cap and body parts being worked at a deviation of less than 1% and being stable in dimension.
- Capsules according to claim 1 characterized in
 that they are made by injection molding of starch or a starch composition having a water content of 5 -25% by weight preferably 17 - 20% by weight calculated to the starch composition.
- Capsules according to any one of claims 1 2
 characterized in that the starch is wheat, potatoe, rice or tapioka starch, or a mixture thereof.
- 4. Capsules according to claim 1 characterized in that they are made by injection molding of gelatin having a water content of 10 20% by weight
 125 calculated to the gelatin composition.
 - 5. Capsules according to any one of claims 1 4 characterized in that the starch and/or the gelatin composition contains one or more plasticizers, extenders, lubricants and/or coloring agents.

- characterized in that at least one part of the open end has the side wall portion recessed with an annular shoulder for receiving and for constituting a stop means in the joining area for the side wall portion at its open end of the other part, both side wall portions being shaped so that the outside surface is smooth in the joining area of said two parts.
- Capsules according to any one of claims 1 6 characterized in that the inner surface of the cap,
 when said cap is mounted on the open end of said body is at practically the same level or below the level of a horizontal plane touching the open end of the cylindrical side wall of said body, whereby, after the filling of the contents and after the closing of said
 cap and body, practically no air is entrapped between said inner surfaces of said cap and the level of said contents.
- Capsules according to claim 7 characterized in that said cap is a circular disc which is coaxially
 joined with the open end of said body by having the plane surface of the circular disc in circumferential engagement with the annular periphery of the open end of said body.
- Capsule according to claim 7 characterized in
 that said cap has an annular recess located at its circumferential edge facing the open end of said body, the side wall portion of said body, mating with and protruding into said recess.
- 10. Capsules according to anyone of claims 1 5 30 characterized in that each of the cap and the body part is die-molded so as to be joined in a distinctive shape to provide a palpable identification of the capsule.
- Capsules according to claim 10 characterized
 in that the capsule parts are molded so as to be formed in a shape adapted to the logogram of its vendor.
 - 12. Capsules according to claim 10 having a shape adapted to its field of application.
- 10 13. Capsules according to anyone of claims 1 -12, characterized in that at least one of the cap and the body part has embossed or debossed printing of letters and/or designs thereon.
- 14. Capsules according to anyone of claims 1 545 and 10 having a shape for its dosage forms.
 - 15. Capsules according to anyone of claims 1 5 and 10 having a shape which provides varying volume of contents.
- 16. Capsules according to anyone of claims 1 550 having a shape which enables a precise positioning on a filling machine.
 - 17. Capsules according to anyone of claims 1 5 having a tablet form.
- 18. Capsules according to anyone of claims 1 17 55 wherein a liquid proof sealing of the cap and the body parts is achieved by wetting the joining surfaces with water or a mixture of water with a water miscible organic solvent.
- A die pressure molded article substantially as
 hereinbefore described with reference to, and as illustrated in the accompanying drawings.

(12) UK Patent Application (19) GB (11) 2 355 269 (13) A

(43) Date of A Publication 18.04.2001

(21) Application No 0019345.8

(22) Date of Filing 08.08.2000

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(51) INT CL7 C11D 17/04 // B65D 65/46

(52) UK CL (Edition S)

C5D DDX D101 D110 D113 D118 D120 D121 D123 D124 D125 D126 D127 D129 D132 D134 D135 D149 D153

D162 D173 D181 D182

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(58) Field of Search

UK CL (Edition S) B8C CWA2 CWP3, C5D D181

INT CL7 B65D 65/46, C11D 17/04 **Online: WPI EPODOC PAJ**

(54) Abstract Title Liquid cleaning composition

(57) A pouched liquid cleaning composition comprises an alkoxylated amine, imine, amide or imide compound, small amount of water and specific levels high ionic strength chelating agents. The pouch is of a water-soluble material such as polyvinyl alcohol.

The alkoxylated compound may be a polyalkoxylated polyamine or polyimine having preferably at least four amine or imine groups and at least four polyalkoxylation groups each having a degree of alkoxylation of at least eight. The chelant may be a phosphonic acid or salt and may include a carboxylic acid or salt. An organic solvent such as a diol compound with glycerine may be present.

Liquid Composition

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Field of Invention

This invention relates to a pouched liquid compositions which comprises an alkoxylated compound having at least two alkoxylated amine, imine, amide or imide groups, small amount of water and specific levels high ionic strength chelating agents.

15 Background to Invention

The laundry industry has been trying to develop ways to make dosing of the detergent products easier. One of method for this is to enclose the detergent in a pouch, such as pouches made from a water-soluble film. Both liquid and solid detergents are known to be delivered to the wash in a easy, safe way. Typically pouch materials are water-soluble films of water-soluble polymers such as derived form polyvinyl alcohol and cellulose.

It is known to incorporate water and other solvents in the compositions in these pouches. One problem associated herewith is that the water-soluble material tends to dissolve in the water in the pouch which weakens the pouch. To address this, the prior art describes that such pouched compositions are to be formulated without any water present. However, the inventors found that one of the problems associated with such compositions in pouches is that these pouches become brittle upon storage. They found that therefor the pouched composition preferably comprises some amount of water and preferably other solvents, to avoid both dissolution and drying

out of the water-soluble material of the pouch and thus to avoid the pouches to become either weak or brittle.

However, the problem with liquid compositions comprising such small amounts of water is phase separation of the ingredients.

Separately, the inventors found also that dissolution of the pouch material can negatively impact the cleaning performance of the ingredients in the composition, resulting in poorer performance in the wash, for example poor clay stain removal.

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To address the problem of poor stain removal, the inventors found that it is advantages to incorporate alkoxylated compounds, having one or more alkoxylated amine, imine, amide or imide groups, preferably polyalkoxylated polyamine or polyimine in the compositions. However, the use of these compounds can result in an even greater phase separation.

The inventors now found surprisingly that when one or more high ionic strength chelating agent are introduced in the composition, the phase separation can be reduced or even avoided, including phase separation of the specific alkoxylated compounds. This thus allows at the same time the use of low amounts of water, resulting in stable pouches and phase stable compositions, and it allows use of the specific alkoxylated compounds without phase separation, resulting in efficient (clay) stain removal.

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This is in particular surprisingly because in isotropic liquid compositions comprising high levels of water, high ionic strength chelating agents can not easily be used, because they tend to crystallise out and cause phase separation. Also, it is generally believed that high ionic strength chelating agents are not compatible with water-soluble material used for the pouches, such as polyvinyl alcohol films.

Summary of Invention

The present invention provides a liquid cleaning composition in a pouch made from a water-soluble material, comprising

- 5 a) a compound having two or more alkoxylated amine, imine, amide or imide groups;
 - b) water, present at a level up to 9% by weight of the composition;
 - c) at least 1.0% by weight of the composition of a chelating agent.

The water-soluble pouch is preferably made of a film comprising PVA.

The water levels in the composition are preferably from 2% to 7.5%; the composition is preferably a laundry composition comprising at least anionic surfactant and fatty acid builder.

The inventors also found that it is in particular beneficial to incorporate in the composition a plasticiser for the water-soluble material of the pouch. They also found that preferably a solvent mixture comprising two or more free hydroxy groups is used

Detailed Description of Invention

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Pouch and Material Thereof

The pouch of the invention, herein referred to as "pouch", is typically a closed structure, made of a water-soluble film described herein, enclosing a volume space which comprises a composition. Said composition is described in more detail hereinafter. The pouch can be of any form, shape and material which is suitable to hold the composition, e.g. without allowing the release of the composition from the pouch prior to contact of the pouch to water. The exact execution will depend on for example, the type and amount of the composition in the pouch, the number of

compartments in the pouch, the characteristics required from the pouch to hold, protect and deliver or release the compositions.

The pouch may have one compartment, holding the liquid composition, or it may have a number of compartment, attached to one another or non-attached to one another, thus having one compartment enclosing (but not attaching) another compartment.

The pouch may be of such a size that it conveniently contains either a unit dose
amount of the composition herein, suitable for the required operation, for example
one wash, or only a partial dose, to allow the consumer greater flexibility to vary the
amount used, for example depending on the size and/or degree of soiling of the wash
load.

It may be preferred that the water soluble film and preferably the pouch as a whole is stretched during formation and/or closing of the pouch, such that the resulting pouch is at least partially stretched. This is to reduce the amount of film required to enclose the volume space of the pouch. When the film is stretched the film thickness decreases. The degree of stretching indicates the amount of stretching of the film by the reduction in the thickness of the film. For example, if by stretching the film, the thickness of the film is exactly halved then the stretch degree of the stretched film is 100%. Also, if the film is stretched so that the film thickness of the stretched film is exactly a quarter of the thickness of the unstretched film then the stretch degree is exactly 200%. Typically and preferably, the thickness and hence the degree of stretching is non-uniform over the pouch, due to the formation and closing process.

Another advantage of using stretching the pouch, is that the stretching action, when forming the shape of the pouch and/or when closing the pouch, stretches the pouch non-uniformly, which results in a pouch which has a non-uniform thickness. This allows control of the dissolution of water-soluble pouches herein, and for example

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sequential release of the components of the detergent composition enclosed by the pouch to the water.

Preferably, the pouch is stretched such that the thickness variation in the pouch formed of the stretched water-soluble film is from 10 to 1000%, preferably 20% to 600%, or even 40% to 500% or even 60% to 400%. This can be measured by any method, for example by use of an appropriate micrometer. Preferably the pouch is made from a water-soluble film that is stretched, said film has a stretch degree of from 40% to 500%, preferably from 40% to 200%.

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The pouch is typically made from a water-soluble film. It is preferred that the pouch as a whole comprises material which is water-dispersible or more preferably watersoluble. Preferred water-soluble films are polymeric materials, preferably polymers which are formed into a film. The material in the form of a film can for example be obtained by casting, blow-moulding, extrusion or blow extrusion of the polymer material, as known in the art.

Preferably the material is water-soluble and has a solubility of at least 50%, preferably at least 75% or even at least 95%, as measured by the method set out hereinafter using a glass-filter with a maximum pore size of 50 microns, namely:

Gravimetric method for determining water-solubility or water-dispersability of the material of the compartment and/or pouch:

50 grams ± 0.1 gram of material is added in a 400 ml beaker, whereof the weight has 25 been determined, and 245ml ± 1ml of distilled water is added. This is stirred vigorously on magnetic stirrer set at 600 rpm, for 30 minutes. Then, the mixture is filtered through a folded qualitative sintered-glass filter with the pore sizes as defined above (max. 50 micron). The water is dried off from the collected filtrate by any conventional method, and the weight of the remaining polymer is determined (which 30

is the dissolved or dispersed fraction). Then, the % solubility or dispersability can be calculated.

Preferred polymer copolymers or derivatives thereof are selected from polyvinyl alcohols, polyalkylene oxides, acrylic acid, cellulose, cellulose ethers, cellulose esters, polyvinyl acetates, polycarboxylic acids and salts, polyaminoacids or peptides, polyamides, copolymers of maleic/acrylic acids, polysaccharides including starch and gelatine, natural gums such as xanthum and carragum. More preferably the polymer is selected from polyacrylates and water-soluble acrylate copolymers, methylcellulose, carboxymethylcellulose sodium, dextrin, ethylcellulose, hydroxyethyl cellulose, hydroxypropyl methylcellulose, maltodextrin, polymethacrylates, most preferably polyvinyl alcohols, polyvinyl alcohol copolymers and hydroxypropyl methyl cellulose (HPMC). Preferably, the level of a type polymer (e.g., commercial mixture) in the film material, for example PVA polymer, is at least 60% by weight of the film.

The polymer can have any weight average molecular weight, preferably from about 1000 to 1,000,000, or even form 10,000 to 300,000 or even form 15,000 to 200,000 or even form 20,000 to 150,000.

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Mixtures of polymers can also be used. This may in particular be beneficial to control the mechanical and/or dissolution properties of the compartment or pouch, depending on the application thereof and the required needs. For example, it may be preferred that a mixture of polymers is present in the material of the compartment, whereby one polymer material has a higher water-solubility than another polymer material, and/or one polymer material has a higher mechanical strength than another polymer material. It may be preferred that a mixture of polymers is used, having different weight average molecular weights, for example a mixture of PVA or a copolymer thereof of a weight average molecular weight of 10,000-40,000, preferably around

20,000, and of PVA or copolymer thereof, with a weight average molecular weight of about 100,000 to 300,000, preferably around 150,000.

Also useful are polymer blend compositions, for example comprising hydrolytically degradable and water-soluble polymer blend such as polylactide and polyvinyl alcohol, achieved by the mixing of polylactide and polyvinyl alcohol, typically comprising 1-35% by weight polylactide and approximately from 65% to 99% by weight polyvinyl alcohol, if the material is to be water-dispersible, or water-soluble.

It may be preferred that the polymer present in the film is from 60-98% hydrolysed, preferably 80% to 90%, to improve the dissolution of the material.

Most preferred are films which are water-soluble and stretchable films, as described above. Highly preferred water-soluble films are films which comprise PVA polymers and that have similar properties to the film known under the trade reference M8630, as sold by Chris-Craft Industrial Products of Gary, Indiana, US.

The water-soluble film herein may comprise other additive ingredients than the polymer or polymer material. For example, it may be beneficial to add plasticisers, for example glycerol, ethylene glycol, diethyleneglycol, propylene glycol, sorbitol and mixtures thereof, additional water, disintegrating aids. It may be useful that the pouch or water-soluble film itself comprises a detergent additive to be delivered to the wash water, for example organic polymeric soil release agents, dispersants, dye transfer inhibitors.

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The pouch is typically process comprising the steps of contacting a composition herein to a water-soluble film in such a way as to partially enclose said composition to obtain a partially formed pouch, optionally contacting said partially formed pouch with a second water-soluble film, and then sealing said partially formed pouch.

Preferably, the pouch is made using a mould, preferably the mould has round inner

side walls and a round inner bottom wall. A composition herein may then be poured into the mould, a second water-soluble film may be placed over the mould with the composition and the pouch may then be sealed, preferably the partially formed pouch is heat sealed. The film is preferably stretched during the formation of the pouch.

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Liquid Composition

Typically the liquid composition is contained in the inner volume space of the pouch, and it may be divided over one or more compartments of the pouch.

- The liquid composition comprises water, up to 9% by weight of the composition, preferably 1% to 8% or even 2% to 7.5% or even 3% to 6% or even 5% by weight of the composition. This is on basis of free water, added to the other ingredients of the composition.
- The liquid composition can made by any method and can have any viscosity, typically depending on its ingredients. The liquid composition preferably has a viscosity of 300 to 3000 cps (centipoises), as measured at a rate of 20 s⁻¹, more preferably from 300 to 2000cps or even from 4000 to 600 cps.
- The liquid composition preferably has a density of 0.8kg/l to 1.3kg/l, preferably around 1.0 to 1.1 kg/l.

The compositions herein are typically cleaning compositions or fabric care compositions, preferably hard surface cleaners, more preferably laundry or dish washing compositions, including pre-treatment or soaking compositions and rinse additive compositions, including fabric enhancers such as softeners, anti-wrinkling agents, perfume compositions. However, preferred are fabric cleaning compositions (laundry detergents).

Alkoxylated amine, imine, amide, imide compound

The composition comprises one or more alkoxylated compounds having at least two alkoxylated amine, imine, amide or imide groups.

5 Preferred are compounds having at least two alkoxylated amine groups.

The alkoxylation group may have one or more alkoxylates, typically more than one, thus forming a chain of alkoxylates, or polyalkoxylation group.

The compound may have two alkoxylation groups or chain, preferably at least 4 or even at least 7 or even at least 10 or even at least 16. Preferred is that the alkoxylation groups are polyalkoxylation groups, (each independently) having an average alkoxylation degree of at least 5, more preferably at least 8, preferably at least 12, up to preferably 80 or even to 50 or even to 25.

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The (poly)alkoxylation is preferably a (poly)ethoxylation and/ or (poly)propoxylation. Thus, preferred is that the alkoxylation group is a polyethoxylation group or polypropoxylation group, or a (poly)ethoxylation/ (poly)propoxyltion group

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Preferred may be that these compounds are polymers having such groups. When used herein an polymer is a compound having 2 or more repeating monomer units forming a backbone. The alkoxylated polymer herein is preferably such that the alkoxylation groups are not part of the backbone of the polymer, but are alkoxylation groups of the amine, imine, amide or imide in the units forming the backbone, or are alkoxylation groups of other side-groups chemically bound to the backbone.

Said alkoxylated compound is preferably a polyamide, polyimide or more preferably a polyamine or polyime compound, whereby these amide, imide, amine or imine units are present as backbone of the polymer, forming the chain of repeating units.

Preferably, these polymers have at least 3 or even 4 or even 5 amide, imide, amine or imine units. Hereby, it may be preferred that only some of the amine or imine are alkoxylated.

It may be preferred that the backbone has also side-chains containing amide, imide, amine or imine groups, which may be alkoxylated.

Preferred are compounds having a weight average molecular weight of 200 to 50,000, preferably to 20,000 or even to 10,000, or even from 350 to 5000 or even to 2000 or even to 1000.

Preferably the composition herein (described in more detail hereinafter) comprises (by weight of the composition) from 0.5% to 15%, more preferably from 0.8% to 10%, more preferably form 1.5% to 8%, more preferably from 2.0% or even 2.5% or even 3% to 6% of said alkoxylated compound. The composition herein may comprise preferably mixtures of the specified compounds.

Preferred backbones, prior to alkoxylation have the general formula:

$$\begin{array}{ccc}
& & & & \downarrow \\
& & & \downarrow \\
[H_2NCH_2CH_2]_n - [NCH_2CH_2]_m - [NCH_2CH_2]_n - NH_2
\end{array}$$

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Highly preferred alkoxylated compounds herein are of the following structures:

$$[H(OCH_{2}CH_{2})_{20}]_{2}N \\ N[(CH_{2}CH_{2}O)_{20}H]_{2} \\ H(OCH_{2}CH_{2})_{20}]_{2}N \\ (CH_{2}CH_{2}O)_{20}H \\ (CH_{2}CH_{2}O)_{20}H \\ N[(CH_{2}CH_{2}O)_{20}H]_{2} \\ N[(CH_{2}CH_{$$

$$[H(OCH_{2}CH_{2})_{7}]_{2}N \\ N[(CH_{2}CH_{2}O)_{7}H]_{2} \\ H(OCH_{2}CH_{2})_{7}N \\ N[(CH_{2}CH_{2}O)_{7}H]_{2} \\ (CH_{2}CH_{2}O)_{7}H \\ N[(CH_{2}CH_{2}O)_{7}H]_{2} \\ (CH_{2}CH_{2}O)_{7}H \\ (CH_{2}CH_{2}O)_{7}H]_{2} \\ N[(CH_{2}CH_{2}O)_{7}H]_{2} \\ N[(CH_{2$$

$$[H(OCH_{2}CH_{2})_{7}]_{2}N \xrightarrow{CH_{3}} N(CH_{2}CH_{2}O)_{7}H \xrightarrow{CH_{3}} N(CH_{2}CH_{2}O)_{7}H$$

$$CH_{3} \times CH_{3} \times CH_{3}$$

Highly preferred are ethoxylated poly(ethyleneimine), preferably having an average ethoxylationd degree per ethoxylation chain of 15 to 25, and a molecular weight of 1000-2000 dalton.

Also highly preferred are ethoxylated tetraethylene pentaimines.

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Chelating agents

The composition herein comprises a chelating agent, typically a high ionic strength chelating agent, having two or more phosphonic acid or phosphonate groups, or two or more carboxylic acid or carboxylate groups, or mixtures thereof. By chelating agent it is meant herein components which act to sequester (chelate) heavy metal ions, but these components may also have calcium and magnesium chelation capacity.

Chelating agents are generally present at a level of from 1%, preferably from 2.5% from 3.5% or even 5.0% or even 7% and preferably up to 20% or even 15% or even 10% by weight of the composition herein.

Highly suitable organic phosphonates herein are amino alkylene poly (alkylene phosphonates), alkali metal ethane 1-hydroxy bisphosphonates and nitrilo trimethylene phosphonates. Preferred among the above species are diethylene triamine penta (methylene phosphonate), ethylene diamine tri (methylene phosphonate) hexamethylene diamine tetra (methylene phosphonate) and hydroxyethylene 1,1 diphosphonate.

Other suitable chelating agents for use herein include nitrilotriacetic acid and polyaminocarboxylic acids such as ethylenediaminotetracetic acid, ethylenetriamine pentacetic acid, ethylenediamine disuccinic acid, ethylenediamine diglutaric acid, 2-hydroxypropylenediamine disuccinic acid or any salts thereof. Especially preferred is ethylenediamine-N,N'-disuccinic acid (EDDS) or the alkali metal, alkaline earth metal, ammonium, or substituted ammonium salts thereof, or mixtures thereof. Glycinamide-N,N'-disuccinic acid (GADS), ethylenediamine-N-N'-diglutaric acid (EDDG) and 2-hydroxypropylenediamine-N-N'-disuccinic acid (HPDDS) are also suitable.

Suitable chelating agents with two or more carboxylates or carboxylic acid groups include the acid or salt forms of succinic acid, malonic acid, (ethylenedioxy) diacetic acid, maleic acid, diglycolic acid, tartaric acid, tartronic acid and fumaric acid, as well as the ether carboxylates and the sulfinyl carboxylates. Chelants containing three carboxy groups include, in particular, the acids or salt forms of citrates, aconitrates and citraconates as well as succinate derivatives. Preferred carboxylate chelants are hydroxycarboxylates containing up to three carboxy groups per molecule, more particularly citrates and citric acids.

Chelating agents containing four carboxy groups include the salts and acid forms of oxydisuccinates, 1,1,2,2-ethane tetracarboxylates, 1,1,3,3-propane tetracarboxylates and 1,1,2,3-propane tetracarboxylates, sulfosuccinate derivatives.

Highly preferred it that at least one organo phosphonate or phosphonic acid and also at least one di- or tri-carboxylate or carboxylic acid is present. Highly preferred is that at least fumaric acid (or salt) and citric acid (or salt) and one or more phosphonates are present. Preferred salts are sodium salts.

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Preferred ingredients of the liquid composition

The preferred amounts of ingredients described herein are % by weight of the composition herein as a whole.

If the liquid composition is a detergent composition, it is preferred that at least a surfactant and builder are present, preferably at least anionic surfactant and preferably also nonionic surfactant, and preferably at least water-soluble builder, preferably at least phosphate builder or more preferably at least fatty acid builder. Preferred is also the presence of enzymes and preferred may also be to incorporate a bleaching agent, such as a preformed peroxyacid.

The liquid composition comprises preferably a colorant or dye and/ or pearlescence agent.

20 Highly preferred are also perfume, brightner, buffering agents (to maintain the pH preferably from 5.5 to 9, more preferably 6 to 8), fabric softening agents, including clays and silicones benefit agents, suds suppressors.

These compounds are described hereinafter in more detail.

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In hard-surface cleaning compositions and dish wash compositions, it is preferred that at least a water-soluble builder is present, such as a phosphate, and preferably also surfactant, perfume, enzymes, bleach.

In fabric enhancing compositions, preferably at least a perfume and a fabric benefit agent are present for example a cationic softening agent, or clay softening agent, anti-wrinkling agent, fabric substantive dye.

Highly preferred in all above compositions are also additional solvents, such as alcohols, diols, monoamine derivatives, glycerol, glycols, polyalkylane glycols, such as polyethylene glycol. Highly preferred are mixtures of solvents, such as mixtures of alcohols, mixtures of diols and alcohols, mixtures. Highly preferred may be that (at least) an alcohol, diol, monoamine derivative and preferably even glycerol are present. The compositions of the invention are preferably concentrated liquids having preferably less than 50% or even less than 40% by weight of solvent, preferably less than 30% or even less than 20% or even less than 35% by weight. Preferably the solvent is present at a level of at least 5% or even at least 10% or even at least 15% by weight of the composition.

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Highly preferred is that the composition comprises, in addition to water, a plasticiser for the water-soluble pouch material, for example one of the plasticisers described above, for example glycerol. Such plasticisers can have the dual purpose of being a solvent for the other ingredients of the composition and a plasticiser for the pouch material.

Anionic Surfactant

The detergent compositions of the invention comprise preferably a surfactant system. Preferably, at least an anionic surfactant is present, preferably at least an sulphonic acid surfactant, such as a linear alkyl benzene sulphonic acid, but salt forms may also be used. Preferably, at least 15% or even at least 20% or even at least 30% by weight of the composition is a surfactant, preferably up to 70% or even 60% or even 50% by weight. Preferably, at least an anionic surfactant and an nonionic surfactant are present in the surfactant system of the composition, preferably in a ratio of 1:2 to 2:1, preferable 1.5:1 to 1:1.5.

The anionic surfactant(s), are preferably present at a level of at least 7.5% by weight of the composition. More preferably anionic surfactant is present at a level of from 10% or even at least 15%, or even from 22.5% by weight of the composition.

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Anionic sulfonate or sulfonic acid surfactants suitable for use herein include the acid and salt forms of a C_5 - C_{20} , more preferably a C_{10} - C_{16} , more preferably a C_{11} - C_{13} alkylbenzene sulfonates, alkyl ester sulfonates, C_6 - C_{22} primary or secondary alkane sulfonates, sulfonated polycarboxylic acids, and any mixtures thereof, but preferably C_{11} - C_{13} alkylbenzene sulfonates.

Anionic sulphate salts or acids surfactants suitable for use in the compositions of the invention include the primary and secondary alkyl sulphates, having a linear or branched alkyl or alkenyl moiety having from 9 to 22 carbon atoms or more preferably C₁₂ to C₁₈ alkyl.

Highly preferred are beta-branched alkyl sulphate surfactants or mixtures of commercial available materials, having a weight average (of the surfactant or the mixture) branching degree of at least 50% or even at least 60% or even at least 80% or even at least 95%. It has been found that these branched sulphate surfactants provide a much better viscosity profile, when clays are present, particular when 5% or more clay is present.

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It may be preferred that the only sulphate surfactant is such a highly branched alkyl sulphate surfactant, namely referred may be that only one type of commercially available branched alkyl sulphate surfactant is present, whereby the weight average branching degree is at least 50%, preferably at least 60% or even at least 80%, or even at least 90%. Preferred is for example Isalchem, as available form Condea.

Mid-chain branched alkyl sulphates or sulfonates are also suitable anionic surfactants for use in the compositions of the invention. Preferred are the mid-chain branched alkyl sulphates. Preferred mid-chain branched primary alkyl sulphate surfactants are of the formula

$$\begin{array}{cccc} & R & R^1 & R^2 \\ & & | & | & | \\ \text{CH}_3\text{CH}_2(\text{CH}_2)_w\text{CH}(\text{CH}_2)_x\text{CH}(\text{CH}_2)_y\text{CH}(\text{CH}_2)_z\text{OSO}_3\text{M} \end{array}$$

These surfactants have a linear primary alkyl sulphate chain backbone (i.e., the longest linear carbon chain which includes the sulphated carbon atom), which preferably comprises from 12 to 19 carbon atoms and their branched primary alkyl moieties comprise preferably a total of at least 14 and preferably no more than 20, carbon atoms. In compositions or components thereof of the invention comprising more than one of these sulphate surfactants, the average total number of carbon atoms for the branched primary alkyl moieties is preferably within the range of from greater than 14.5 to about 17.5. Thus, the surfactant system preferably comprises at least one branched primary alkyl sulphate surfactant compound having a longest linear carbon chain of not less than 12 carbon atoms or not more than 19 carbon atoms, and the total number of carbon atoms including branching must be at least 14, and further the average total number of carbon atoms for the branched primary alkyl moiety is within the range of greater than 14.5 to about 17.5.

Preferred mono-methyl branched primary alkyl sulphates are selected from the group consisting of: 3-methyl pentadecanol sulphate, 4-methyl pentadecanol sulphate, 5-methyl pentadecanol sulphate, 6-methyl pentadecanol sulphate, 7-methyl pentadecanol sulphate, 8-methyl pentadecanol sulphate, 9-methyl pentadecanol sulphate, 10-methyl pentadecanol sulphate, 11-methyl pentadecanol sulphate, 12-methyl pentadecanol sulphate, 13-methyl pentadecanol sulphate, 3-methyl hexadecanol sulphate, 4-methyl hexadecanol sulphate, 5-methyl hexadecanol sulphate, 6-methyl hexadecanol sulphate, 7-methyl hexadecanol sulphate, 8-methyl hexadecanol sulphate, 9-methyl hexadecanol sulphate, 10-methyl hexadecanol

sulphate, 11-methyl hexadecanol sulphate, 12-methyl hexadecanol sulphate, 13-methyl hexadecanol sulphate, 14-methyl hexadecanol sulphate, and mixtures thereof.

Preferred di-methyl branched primary alkyl sulphates are selected from the group consisting of: 2,3-methyl tetradecanol sulphate, 2,4-methyl tetradecanol sulphate, 2,5-methyl tetradecanol sulphate, 2,6-methyl tetradecanol sulphate, 2,7-methyl tetradecanol sulphate, 2,9-methyl tetradecanol sulphate, 2,9-methyl tetradecanol sulphate, 2,10-methyl tetradecanol sulphate, 2,11-methyl tetradecanol sulphate, 2,12-methyl tetradecanol sulphate, 2,3-methyl pentadecanol sulphate, 2,4-methyl pentadecanol sulphate, 2,5-methyl pentadecanol sulphate, 2,6-methyl pentadecanol sulphate, 2,7-methyl pentadecanol sulphate, 2,8-methyl pentadecanol sulphate, 2,9-methyl pentadecanol sulphate, 2,11-methyl pentadecanol sulphate, 2,11-methyl pentadecanol sulphate, 2,11-methyl pentadecanol sulphate, 2,12-methyl pentadecanol sulphate, 2,13-methyl pentadecanol sulphate, and mixtures thereof.

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It is preferred that the anionic surfactants herein are present in the form of sodium salts.

Nonionic alkoxylated surfactant

Essentially any alkoxylated nonionic surfactants, which is not an alkoxylated amine, imine, amide or imide compound of the invention, can be comprised by the composition herein. Thus, these nonionic surfactants are then present in addition to the alkoxylated polymer compound of the invention. Ethoxylated and propoxylated nonionic surfactants are preferred. Preferred alkoxylated surfactants can be selected from the classes of the nonionic condensates of alkyl phenols, nonionic ethoxylated alcohols, nonionic ethoxylated/propoxylated fatty alcohols.

Highly preferred are nonionic alkoxylated alcohol surfactants, being the condensation products of aliphatic alcohols with from 1 to 75 moles of alkylene oxide, in particular about 50 or from 1 to 15 moles, preferably to 11 moles,

particularly ethylene oxide and/or propylene oxide, are highly preferred nonionic surfactants. The alkyl chain of the aliphatic alcohol can either be straight or branched, primary or secondary, and generally contains from 6 to 22 carbon atoms. Particularly preferred are the condensation products of alcohols having an alkyl group containing from 8 to 20 carbon atoms with from 2 to 9 moles and in particular 3 or 5 moles, of ethylene oxide per mole of alcohol.

Polyhydroxy fatty acid amides are highly preferred nonionic surfactant comprised by the composition, in particular those having the structural formula R^2CONR^1Z wherein: R1 is H, C_{1-18} , preferably C_1 -C4 hydrocarbyl, 2-hydroxy ethyl, 2-hydroxy propyl, ethoxy, propoxy, or a mixture thereof, preferable C1-C4 alkyl, more preferably C_1 or C_2 alkyl, most preferably C_1 alkyl (i.e., methyl); and R_2 is a C_5 - C_{31} hydrocarbyl, preferably straight-chain C_5 - C_{19} or C_7 - C_{19} alkyl or alkenyl, more preferably straight-chain C_9 - C_{17} alkyl or alkenyl, most preferably straight-chain C_{11} - C_{17} alkyl or alkenyl, or mixture thereof; and Z is a polyhydroxyhydrocarbyl having a linear hydrocarbyl chain with at least 3 hydroxyls directly connected to the chain, or an alkoxylated derivative (preferably ethoxylated or propoxylated) thereof. Z preferably will be derived from a reducing sugar in a reductive amination reaction; more preferably Z is a glycityl.

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Cationic surfactant

Preferred are also cationic mono-alkoxylated and bis-alkoxylated quaternary amine surfactants with a C_6 - C_{18} N-alkyl chain, such as of the general formula I:

wherein R¹ is an alkyl or alkenyl moiety containing from about 6 to about 18 carbon atoms, preferably 6 to about 16 carbon atoms, most preferably from about 6 to about 14 carbon atoms; R² and R³ are each independently alkyl groups containing from one to about three carbon atoms, preferably methyl, most preferably both R² and R³ are methyl groups; R⁴ is selected from hydrogen (preferred), methyl and ethyl; X⁻ is an anion such as chloride, bromide, methylsulphate, sulphate, or the like, to provide electrical neutrality; A is a alkoxy group, especially a ethoxy, propoxy or butoxy group; and p is from 0 to about 30, preferably 2 to about 15, most preferably 2 to about 8.

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The cationic bis-alkoxylated amine surfactant preferably has the general formula II:

$$R^{1}$$
 ApR^{3}
 X^{-}
 R^{2}
 $A'qR^{4}$
(II)

wherein R^1 is an alkyl or alkenyl moiety containing from about 8 to about 18 carbon atoms, preferably 10 to about 16 carbon atoms, most preferably from about 10 to about 14 carbon atoms; R^2 is an alkyl group containing from one to three carbon atoms, preferably methyl; R^3 and R^4 can vary independently and are selected from hydrogen (preferred), methyl and ethyl, X^- is an anion such as chloride, bromide, methylsulphate, sulphate, or the like, sufficient to provide electrical neutrality. A and A' can vary independently and are each selected from C_1 - C_4 alkoxy, especially ethoxy, (i.e., $-CH_2CH_2O_-$), propoxy, butoxy and mixtures thereof; p is from 1 to about 30, preferably 1 to about 4 and q is from 1 to about 30, preferably 1 to about 4, and most preferably both p and q are 1.

Another suitable group of cationic surfactants which can be used in the detergent compositions are cationic ester surfactants. Suitable cationic ester surfactants,

including choline ester surfactants, have for example been disclosed in US Patents No.s 4228042, 4239660 and 4260529.

5 Builder compounds

The compositions in accord with the present invention preferably contain a water-soluble builder compound, typically present in detergent compositions at a level of from 1% to 60% by weight, preferably from 3% to 40% by weight, most preferably from 5% to 25% by weight of the composition.

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Suitable water-soluble builder compounds include the water soluble monomeric carboxylates, or their acid forms, or homo or copolymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxylic radicals separated from each other by not more that two carbon atoms, and mixtures of any of the foregoing.

Highly preferred maybe that one or more fatty acids and/ or optionally salts thereof (and then preferably sodium salts) are present in the detergent composition. It has been found that this can provide further improved softening and cleaning of the fabrics. Preferably, the compositions contain 1% to 25% by weight of a fatty acid or salt thereof, more preferably 6% to 18% or even 10% to 16% by weight. Preferred are in particular C_{12} - C_{18} saturated and/ or unsaturated fatty acids, but preferably mixtures of such fatty acids. Highly preferred have been found mixtures of saturated and unsaturated fatty acids, for example preferred is a mixture of rape seed-derived fatty acid and C_{16} - C_{18} topped whole cut fatty acids, or a mixture of rape seed-derived fatty acid and a tallow alcohol derived fatty acid.

The detergent compositions of the invention may comprise phosphate-containing builder material. Preferably present at a level of from 2% to 40%, more preferably from 3% to 30%, more preferably from 5% to 20%. Suitable examples of water-

soluble phosphate builders are the alkali metal tripolyphosphates, sodium, potassium and ammonium pyrophosphate, sodium and potassium and ammonium pyrophosphate, sodium and potassium orthophosphate, sodium polymeta/phosphate in which the degree of polymerization ranges from about 6 to 21, and salts of phytic acid.

The compositions in accord with the present invention may contain a partially soluble or insoluble builder compound, typically present in detergent compositions at a level of from 0.5% to 60% by weight, preferably from 5% to 50% by weight, most preferably from 8% to 40% weight of the composition.

Preferred are aluminosilicates and/ or crystalline layered silicates such as SKS-6, available from Clariant.

However, from a formulation point of view it may be preferred not to include such builders in the liquid composition, because it will lead to too much dispersed or precipitate material in the liquid, or it requires too much process or dispersion aids.

Perfume

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20 Highly preferred are perfume components, preferably at least one component comprising a coating agent and/ or carrier material, preferably organic polymer carrying the perfume or alumniosilicate carrying the perfume, or an encapsulate enclosing the perfume, for example starch or other cellulosic material encapsulate.

The inventors have found that the perfumes are more efficiently deposited onto the fabric in the compositions of the invention.

Fabric softening clays

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Preferred fabric softening clays are smectite clays, which can also be used to prepare the organophilic clays described hereinafter, for example as disclosed in EP-A- 299575 and EP-A-313146. Specific examples of suitable smectite clays are selected from the classes of the bentonites- also known as montmorillonites, hectorites, volchonskoites, nontronites, saponites and sauconites, particularly those having an alkali or alkaline earth metal ion within the crystal lattice structure.

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Preferably, hectorites or montmorillonites or mixtures thereof. Hectorites are most preferred clays.

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The softening clay is preferably present at levels up to 15%, more preferably up to 7% or even up to 10% by weight, typically, at least 3% or even at least 5%. By weight, whent he formulation is to be a softening formulation.

The hectorite clays suitable in the present composition should preferably be sodium clays, for better softening activity.

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Sodium clays are either naturally occurring, or are naturally-occuring calcium-clays which have been treated so as to convert them to sodium-clays. If calcium-clays are used in the present compositions, a salt of sodium can be added to the compositions in order to convert the calcium clay to a sodium clay. Preferably, such a salt is sodium carbonate, typically added at levels of up to 5% of the total amount of clay.

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Examples of hectorite clays suitable for the present compositions include Bentone EW as sold by Elementis.

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Another preferred clay is an organophilic clay, preferably a smectite clay, whereby at least 30% or even at least 40% or preferably at least 50% or even at least 60% of the exchangeable cations is replaced by a, preferably long-chain, organic cations. Such clays are also referred to as hydrophobic clays. The cation exchange capacity of clays and the percentage of exchange of the cations with the long-chain organic cations can be measured in several ways known in the art, as for example fully set out in

Grimshaw, The Chemistry and Physics of Clays, Interscience Publishers, Inc.,pp. 264-265 (1971).

Whilst the organophilic smectite clay provides excellent softening benefit, they can increase the viscosity of the liquid compositions. Therefore, it will depend on the viscosity requirements of the composition, how much of these organophlic clays can be used. Typically, they are used in the liquid detergent compositions of the invention at a level of from 0.1% to 10%, more preferably from 0.3% to 7%, most preferably from 0.4% to 5% or even 0.5% to 4% by weight of the composition.

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These organophilic clays are formed prior to incorporation into the detergent composition. Thus for example, the cations, or part thereof, of the normal smectite clays are replaced by the long-chain organic cations to form the organophilic smectite clays herein, prior to further processing of the material to form the detergents of the invention.

The organophilic clay is preferably in the form of a platelet or lath-shaped particle. Preferably the ratio of the width to the length of such a platelet is at least 1:2, preferably at least 1:4 or even at least 1:6 or even at least 1:8.

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When used herein, a long-chain organic cation can be any compound which comprises at least one chain having at least 6 carbon atoms, but typically at least 10 carbon atoms, preferably at least 12 carbon atoms, or in certain embodiments of the invention, at least 16 or even at least 18 carbon atoms. Preferred long-chain organic cations are described hereinafter.

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Preferred organophilic clays herein clay are smectite clays, preferably hectorite clays and/ or montmorillonite clays containing one or more organic cations of formulae:

$$R_{4} = N^{+} = R_{2}$$
 R_{3}
 $R_{6}(CH_{2})n = N$
 $N^{+} = CH_{3}$

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where R₁ represents an organic radical selected from R₇, R₇-CO-O-(CH₂)_n, or

 R_7 -CO-NR₈- in which R_7 is an alkyl, alkenyl or alkylaryl group with 12-22 carbon atoms, whereby R_8 is hydrogen, C_1 - C_4 alkyl, alkenyl or hydroxyalkyl, preferably - CH_3 or - C_2H_5 or -H; n is an integer, preferably equal to 2 or 3; R_2 represents an organic radical selected from R_1 or C_1 - C_4 alkyl, alkenyl or hydroxyalkyl, preferably - CH_3 or - CH_2CH_2OH ; R_3 and R_4 are organic radicals selected from C_1 - C_4 alkyl-aryl, C_1 - C_4 alkyl, alkenyl or hydroxyalkyl, preferably - CH_3 , - CH_2CH_2OH , or benzyl group; R_5 is an alkyl or alkenyl group with 12-22 carbon atoms; R_6 is preferably -OH, - NHCO- R_7 , or -OCO- R_7 .

Highly preferred cations are quaternary ammonium cations having two C_{16} - C_{28} or even C_{16} - C_{24} alkyl chains. Highly preferred are one or more organic cations which have one or preferably two alkyl groups derived from natural fatty alcohols, the cations preferably being selected from dicocoyl methyl benzyl ammonium, dicocoyl ethyl benzyl ammonium, dicocoyl dimethyl ammonium, dicocoyl diethyl ammonium; more preferably ditallow diethyl ammonium, ditallow ethyl benzyl ammonium; more preferably ditallow dimethyl ammonium and/ or ditallow methyl benzyl ammonium.

It may be highly preferred that mixtures of organic cations are present.

Highly preferred are organophilic clays as available from Rheox/Elementis, such as Bentone SD-1 and Bentone SD-3, which are registered trademarks of Rheox/Elementis.

Cationic fabric softening agents

Cationic fabric softening agents are preferably present in the composition herein. Suitable cationic fabric softening agents include the water insoluble tertiary amines or dilong chain amide materials as disclosed in GB-A-1 514 276 and EP-B-0 011 340. Preferably, these water-insoluble tertiary amines or dilong chain amide materials are comprised by the solid component of the composition herein.

Cationic fabric softening agents are typically incorporated at total levels of from 0.5% to 15% by weight, normally from 1% to 5% by weight.

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Bleaching agent

Another ingredient which may be present is a perhydrate bleach, such as salts of percarbonates, particularly the sodium salts, and/ or organic peroxyacid bleach precursor. It has been found that when the pouch or compartment is formed from a material with free hydroxy groups, such as PVA, the preferred bleaching agent comprises a percarbonate salt and is preferably free form any perborate salts or borate salts. It has been found that borates and perborates interact with these hydroxy-containing materials and reduce the dissolution of the materials and also result in reduced performance.

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Inorganic perhydrate salts are a preferred source of peroxide. Preferably these salts are present at a level of from 0.01% to 50% by weight, more preferably of from 0.5% to 30% by weight of the composition or component.

Examples of inorganic perhydrate salts include percarbonate, perphosphate, persulfate and persilicate salts. The inorganic perhydrate salts are normally the alkali metal salts. The inorganic perhydrate salt may be included as the crystalline solid without additional protection. For certain perhydrate salts however, the preferred executions of such granular compositions utilise a coated form of the material which provides better storage stability for the perhydrate salt in the granular product.

Suitable coatings comprise inorganic salts such as alkali metal silicate, carbonate or borate salts or mixtures thereof, or organic materials such as waxes, oils, or fatty soaps.

- Alkali metal percarbonates, particularly sodium percarbonate are preferred perhydrates herein. Sodium percarbonate is an addition compound having a formula corresponding to 2Na₂CO₃.3H₂O₂, and is available commercially as a crystalline solid.
- The composition herein preferably comprises a peroxy acid or a precursor therefor (bleach activator), preferably comprising an organic peroxyacid bleach precursor. It may be preferred that the composition comprises at least two peroxy acid bleach precursors, preferably at least one hydrophobic peroxyacid bleach precursor and at least one hydrophilic peroxy acid bleach precursor, as defined herein. The production of the organic peroxyacid occurs then by an in situ reaction of the precursor with a source of hydrogen peroxide.

The hydrophobic peroxy acid bleach precursor preferably comprises a compound having a oxy-benzene sulphonate group, preferably NOBS, DOBS, LOBS and/ or NACA-OBS, as described herein.

The hydrophilic peroxy acid bleach precursor preferably comprises TAED, as described herein.

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Amide substituted alkyl peroxyacid precursor compounds are suitable herein, including those of the following general formulae:

$$R^{1} - C - N - R^{2} - C - L$$
 $R^{1} - N - C - R^{2} - C - L$
O R^{5} O or R^{5} O O

wherein R¹ is an alkyl group with from 1 to 14 carbon atoms, R² is an alkylene group containing from 1 to 14 carbon atoms, and R⁵ is H or an alkyl group containing 1 to 10 carbon atoms and L can be essentially any leaving group. Amide substituted bleach activator compounds of this type are described in EP-A-0170386.

The composition may contain a pre-formed organic peroxyacid.

A preferred class of organic peroxyacid compounds are the amide substituted compounds of the following general formulae:

$$R^{1}$$
 — C — N — R^{2} — C — OOH R^{1} — N — C — R^{2} — C — OOH R^{5} O Or R^{5} O O

wherein R¹ is an alkyl, aryl or alkaryl group with from 1 to 14 carbon atoms, R² is an alkylene, arylene, and alkarylene group containing from 1 to 14 carbon atoms, and R⁵ is H or an alkyl, aryl, or alkaryl group containing 1 to 10 carbon atoms. Amide substituted organic peroxyacid compounds of this type are described in EP-A-0170386.

Other organic peroxyacids include diacyl and tetraacylperoxides, especially
diperoxydodecanedioc acid, diperoxytetradecanedioc acid and
diperoxyhexadecanedioc acid. Mono- and diperazelaic acid, mono- and
diperbrassylic acid and N-phthaloylaminoperoxicaproic acid are also suitable herein.

Suds suppressing system

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The composition may comprise a suds suppresser at a level less than 10%, preferably 0.001% to 10%, preferably from 0.01% to 8%, most preferably from 0.05% to 5%, by weight of the composition Preferably the suds suppresser is either a soap, paraffin, wax, or any combination thereof. If the suds suppresser is a suds suppressing

silicone, then the detergent composition preferably comprises from 0.005% to 0.5% by weight a suds suppressing silicone.

Enzymes

Another preferred ingredient useful in the compositions herein is one or more enzymes.

Preferred enzymatic materials include the commercially available lipases, cutinases, amylases, neutral and alkaline proteases, cellulases, endolases, esterases, pectinases, lactases and peroxidases conventionally incorporated into detergent compositions. Suitable enzymes are discussed in US Patents 3,519,570 and 3,533,139.

Preferred commercially available protease enzymes include those sold under the tradenames Alcalase, Savinase, Primase, Durazym, and Esperase by Novo Industries A/S (Denmark), those sold under the tradename Maxatase, Maxacal and Maxapem by Gist-Brocades, those sold by Genencor International, and those sold under the tradename Opticlean and Optimase by Solvay Enzymes. Protease enzyme may be incorporated into the compositions in accordance with the invention at a level of from 0.0001% to 4% active enzyme by weight of the composition.

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Preferred amylases include, for example, α-amylases obtained from a special strain of B licheniformis, described in more detail in GB-1,269,839 (Novo). Preferred commercially available amylases include for example, those sold under the tradename Rapidase by Gist-Brocades, and those sold under the tradename Termamyl, Duramyl and BAN by Novo Industries A/S. Highly preferred amylase enzymes maybe those described in PCT/ US 9703635, and in WO95/26397 and WO96/23873.

Amylase enzyme may be incorporated into the composition in accordance with the invention at a level of from 0.0001% to 2% active enzyme by weight of the composition.

Useful additional non-alkoxylated organic polymeric compounds for inclusion in the compositions herein include the water soluble organic homo- or co-polymeric polycarboxylic acids or their salts in which the polycarboxylic acid comprises at least two carboxyl radicals separated from each other by not more than two carbon atoms. Polymers of the latter type are disclosed in GB-A-1,596,756. Examples of such salts are polyacrylates of MWt 1000-5000 and their copolymers with maleic anhydride, such copolymers having a molecular weight of from 2000 to 100,000, especially 40,000 to 80,000.

Other organic polymeric compounds suitable for incorporation in the detergent compositions herein include cellulose derivatives.

Suitable suds suppressing systems for use herein may comprise essentially any known antifoam compound, including for example silicone antifoam compounds and 2-alkyl alcanol antifoam compounds.

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Other suitable antifoam compounds include the monocarboxylic fatty acids and soluble salts thereof, as also described as builders above. These materials are described in US Patent 2,954,347, issued September 27, 1960 to Wayne St. John. The monocarboxylic fatty acids, and salts thereof, for use as suds suppressor typically have hydrocarbyl chains of 10 to 24 carbon atoms, preferably 12 to 18 carbon atoms. Suitable salts include the alkali metal salts such as in particular sodium but also potassium salts.

The compositions herein may also comprise from 0.01% to 10 %, preferably from 0.05% to 0.5% by weight of polymeric dye transfer inhibiting agents. The polymeric

dye transfer inhibiting agents are preferably selected from polyamine N-oxide polymers, copolymers of N-vinylpyrrolidone and N-vinylimidazole, polyvinylpyrrolidonepolymers or combinations thereof, whereby these polymers can be cross-linked polymers.

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The compositions herein also optionally contain from about 0.005% to 5% by weight of certain types of hydrophilic optical brighteners.

Preferred brighteners include 4,4',-bis[(4-anilino-6-(N-2-bis-hydroxyethyl)-s-triazine-2-yl)amino]-2,2'-stilbenedisulfonic acid and disodium salt, commercially marketed under the tradename Tinopal-UNPA-GX by Ciba-Geigy Corporation; 4,4'-bis[(4-anilino-6-(N-2-hydroxyethyl-N-methylamino)-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid disodium salt, commercially marketed under the tradename Tinopal 5BM-GX by Ciba-Geigy Corporation; 4,4'-bis[(4-anilino-6-morphilino-s-triazine-2-yl)amino]2,2'-stilbenedisulfonic acid, sodium salt, commercially marketed under the tradename Tinopal-DMS-X and Tinopal AMS-GX by Ciba Geigy Corporation.

Also preferred may be bleaches, neutralizing agents, buffering agents, phase regulants, hydrotropes, enzyme stabilizing agents, opacifiers, anti-oxidants, bactericides, photo-bleaches.

Abbreviations used in Examples

In the detergent compositions, the abbreviated component identifications have the following meanings:

LAS

Sodium linear C₁₁₋₁₃ alkyl benzene sulfonate

HSAS

C₁₁₋₁₃ alkyl benzene sulfonic acid

30 TAS

Sodium tallow alkyl sulphate

~ \ . ~		G 1' G	O 11 1	11.
('VUA'	•	Sodium L'i	.[`1 2][[7][[7]	ilnhate
CxyAS	•	Sodium C_{1x}	· C V alkyl st	arpriace

CxyASz: Sodium C_{1x} - C_{1y} alkyl sulphate, having a weight

average branching degree of at least z

QAS : $R_2.N^+(CH_3)_2(C_2H_4OH)$ with $R_2 = C_{12} - C_{14}$

5 MBAS : Branched C16-C18 alkylsulphate having an average

methyl branching of 1.5

APA : C₈ - C₁₀ amido propyl dimethyl amine

Soap : Sodium linear alkyl carboxylate derived from an

80/20 mixture of tallow and coconut fatty acids

10 TPKFA : C₁₆₋C₁₈ topped whole cut fatty acids

Citric acid : Anhydrous citric acid

Citrate: Tri-sodium citrate dihydrate of activity 86.4% with a

particle size distribution between 425µm and 850µm

MA/AA : Copolymer of 1:4 maleic/acrylic acid, average

molecular weight about 70,000

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Cellulose ether : Methyl cellulose ether with a degree of

polymerization of 650 available from Shin Etsu

Chemicals

EA I : Ethoxylated tetraethylene pentaimine having at least 5

ethoxylation groups, each having an average

ethoxylation degree of 15-25.

EA II : Ethoxylated imine having at least 10 ethoxylated

amine groups, each having an average ethoxylation

degree of 15-25.

25 Protease : Proteolytic enzyme, having 3.3% by weight of active

enzyme, sold by NOVO Industries A/S under the

tradename Savinase

Callulaga		Cellulytic enzyme,	having 0.23% by	v vieight of active
Cellulase	;	Centulylic enzyme,	naving 0.23% by	weight of active

enzyme, sold by NOVO Industries A/S under the

tradename Carezyme

Amylase : Amylolytic enzyme, having 1.6% by weight of active

enzyme, sold by NOVO Industries A/S under the

tradename Termamyl 120T

Lipase : Lipolytic enzyme, having 2.0% by weight of active

enzyme, sold by NOVO Industries A/S under the

tradename Lipolase or Lipolase Ultra

10 : Sodium percarbonate of nominal formula

2Na₂CO₃.3H₂O₂

EDDS : Ethylenediamine-N,N'-disuccinic acid, (S,S) isomer in

the form of its sodium salt.

HEDP: 1,1-hydroxyethane diphosphonic acid

15 Photoactivated bleach: Sulfonated zinc phthlocyanine encapsulated in dextrin

soluble polymer

Brightener : Disodium 4,4'-bis(2-sulphostyryl)biphenyl or

Disodium 4,4'-bis(4-anilino-6-morpholino-1.3.5-

triazin-2-yl)amino) stilbene-2:2'-disulfonate

20 PVNO : Polyvinylpyridine N-oxide polymer, with an average

molecular weight of 50,000

PVPVI : Copolymer of polyvinylpyrolidone and

vinylimidazole, with an average molecular weight of

20,000

25 Clay : Bentone SD-3, as available from Rheox/Elementis,

and/or Bentone EW, as available from

Rheox/Elementis

Opacifier

In the following examples all levels are quoted as % by weight of the composition:

Example 1

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The following liquid composition is in accord with the invention, and is incorporated in a polyvinyl alcohol film, of monosol material.

Component	<u>A</u>	<u>B</u>	<u>C</u>
C25AS		20.0	1
HLAS (acid)	25.0	5.0	19.0
Citric Acid	4.0	4.5	3.0
C ₁₂₋₁₈ Fatty Acid (s)	16.0	22.0	18.0
Enzymes	1.3	1.0	0.5
QAS	1.0	1.0	-
Propanediol/glycerol/ethanol	26	30	27.0
Formic acid	1.0	-	-
APA	3.0	-	15.0
HEDP or EDDS	2.0	0.8	1.25
Clay	5.0	•	***
Polymer I	2.6	3.0	•
Polymer II	2.0	1.5	4.0
Fumaric acid	3.0	2.0	2.0
Dye, Perfume, Brighteners,	Balance	Balance	<u>Balan</u>
Preservatives, Suds Suppressor,			<u>ce</u>
Other Minors, water (N4%)			
	100%	100%	100%

Example 2

5 The following liquid detergent formulations are according to the present invention and incorporated in a polyvinyl alcohol film of monosol material.

	A	В	C
HLAS or LAS	2.0	16.0	-
MBAS	-	1.8	18.0
C25AS		12.0	4.0
rape seed oil fatty acid	3.5	5.0	•
APA	20.0	-	10.0
TPKFA	6.0	15.0	18.0
Citric acid	2.0	3.0	4.01
Glcerol	-	10.0	10.0
Ethanol	4.0	8.0	_
1,2 Propanediol	10.0	4.0	12.0
Monoethanolamine	3.0	5.0	4.0
CMC	-	-	0.5
Clay	8.0	-	6.0
HEDPP/EDDS	2.0	2.0	1.0
Polymer I	2.0	3.0	-
Polymer II	2.0	2.0	5.0
Protease	0.2	1.0	0.9
Lipase	-	-	0.5
Amylase	0.4	0.5	0.3
Cellulase	-	0.8	-

Fumate	3.0	2.0	1.0
Ca chloride	1.0	.05	0.5
Brightener 1	0.5	0.4	0.5
Suds suppressor	0.5	0.5	0.8
Perfume	1.0	1.0	1.0
Opacifier	0.5	-	0.5
Minors up to 100%			
NaOH up to pH 8	-	_	-
water up to balance			

Example 3

The following are compositions in accord with the invention.

	A	В	С	
C2 _y AE5	14.0	20.0	10.0	
MBAS/HLAS	5.0	-	-	
Citric acid/ sodium	5.0	10.0	8.0	
citrate				
Phosphate	5.0	10.0	5.0	
Fatty acid	5.0	10.0	5.0	
Hydrochlorid acid	10.0	-	-	
Polymer I	3.0	6.0	3.0	
Polymer II	5.0	2.0	3.0	
Peroxyacid bleach	-	15.0	15.0	
Glycerol	5.0	7.0	10.0	
Ethanol	15.0	9.0	20.0	
Monoethandamine	15.0	-	5.0	
EDDS/HEDP	5.0	4.0	3.0	
Water	6.0	5.0	4.0	
Fumate	-	-	6.0	
Perfume	1.0	0.5	1.0	
enzymes	-	-	1.5	
Dye	10ppm	10ppm	50ppm	
Pearlescence	0.1	0.2	0.5	
Minors to balance to 100%				

Claims

- 1. A liquid cleaning composition in a pouch made from a water-soluble material, comprising
- a) a compound having two or more alkoxylated amine, imine, amide or imide groups;
 - b) water, present at a level up to 9% by weight;
 - c) at least 1.0% by weight of a chelating agent.
- 10 2. A composition according to claim 1, whereby said water-soluble material is a film comprising polyvinyl alcohol.
 - 3. A composition according to claims 1 or 2, whereby said alkoxylated compound is a polyalkoxylated polyamine or polyimine compound, preferably a polyethoxylated and/ or polypropoxylated polyamine or polyimine compound.
 - 4. A composition as in claim 3 whereby said compound comprises at least 4 amine or imine groups and at least 4 polyalkoxylation groups each having an average degree of alkoxylation of at least 8, preferably 12 to 50 or even to 25.
 - 5. A composition as in any preceding claim whereby the composition comprises at least 2.5% by weight of phosphonic acid or salt chelating agent and optionally carboxylic acid or salt chelating agent, preferably at least 3.5% by weight.
- 6. A composition as in any preceding claim comprising an organic solvent mixture comprising at least a compound with one or more free hydroxy groups and/ or a plasticiser for the water-soluble pouch material, the composition preferably comprising from 2% to 7.5% water.

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- 7. A composition according to claim 6 whereby the organic solvent mixture comprises an diol-compound and glycerine, preferably at a total level of at least 10% by weight of the composition.
- 5 8. A composition as in any preceding claim comprising at least 15% of water-soluble builder including fatty acid.
 - 9. A composition as in any preceding claim whereby the surfactant system is present at a level of at least 30% by weight of the composition and comprises at least an anionic surfactant and an nonionic surfactant, in a ratio of 1:2 to 2:1, preferable 1.5:1 to 1:1.5.
 - 10. A composition according to any preceding claim, whereby said water-soluble film is stretchable, said water-soluble film has a stretch degree from 40% to 500%.
 - 11. A composition according to any preceding claim comprising a bleaching agent and/ or enzyme.

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Application No:

GB 0019345.8

Claims searched: 1-11 Examiner:

Michael Conlon

Date of search:

31 January 2001

Patents Act 1977 Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.S): C5D D181 B8C CWP3 CWA2

Int Cl (Ed.7): B65D 65/46 Cl1D 17/04

Online: WPI EPODOC PAJ Other:

Documents considered to be relevant:

Category	Identity of document and relevant passage			
Α	US6136776	(Dickler) Exx 14 and 11	1	
A	US4610799	(Wilsberg) Claim 2	1	
A	Derwent Abstract N	No. 1995-063972 [09] relating to JP060340899 (Kao)	Į.	

Document indicating lack of novelty or inventive step

one or more other documents of same category.

Document indicating lack of inventive step if combined with

Member of the same patent family

Document indicating technological background and/or state of the art.

Document published on or after the declared priority date but before the filing date of this invention.

Patent document published on or after, but with priority date earlier than, the filing date of this application.

(19)日本国特許庁 (JP)

(12) 公開特許公報(A)

(11)特許出願公開番号

特開平10-338251

(43)公開日 平成10年(1998)12月22日

(51) Int.Cl. ⁶		記号	FΙ		
B65D	41/04		B65D	41/04	C
	47/08			47/08	H

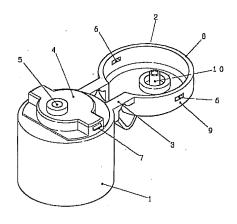
審査請求 未請求 請求項の数1 書面 (全 4 頁)

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(54)【発明の名称】 ヒンジ付きキャップ

(57)【要約】

【課題】 蓋の開閉が簡単でしかも不用意に開蓋しない合成樹脂製のヒンジ付きキャップを安価で提供する。 【解決手段】 蓋2の側面部に可撓性を有し、キャップ本体1の天面4の両横側及び蓋2の両横側面9に係合部を設け、閉蓋時には各々の係合部が係合することでロックされ、開蓋時には蓋2の前側面8を押圧することで蓋2の側面が撓み、横側面9が広げられて係合が解かれることでロックが解除されるようにしたので、蓋2を天面4へ向けて押さ込込むだけで、閉蓋と同時に蓋2を容易にロックすることができるうえ、前側面8を前方に押圧しながら上方に引き上げることによって簡単に蓋2が開けられ、ロック機構を解除するための煩わしい操作を必要とせず、しかも不用意に蓋2が開いてしまうことがない。



【特許請求の範囲】

【請求項1】天面に注液口を有し、側面に容器へ係止される係止筒部を有するキャップ本体と、該キャップ本体の天面を覆い、後側に設けられたヒンジを回転軸として開閉可能な蓋とからなり、閉蓋状態ではロックされ、開蓋時にはロックの解除が自在なロック機構を有するヒンジ付きキャップにおいて、蓋の側面部に可撓性を有し、キャップ本体の天面の両横側及び蓋の両横側面に係合部を設け、閉蓋時には各々の係合部が係合することでロックされ、開蓋時には整の前側面を押圧することで蓋の側面が携み、横側面が広がって係合が解かれることでロックが解除み、横側面が広がって係合が解かれることでロックが解除されるようにしたことを特徴とするヒンジ付きキャップ。

【発明の詳細な説明】

[0001]

【発明の属する技術分野】この発明は、薬液等を封入して適宜注液、塗布を行うための容器に取り付けられるヒンジ付きキャップに関するものであり、特に、蓋の開閉が簡単でしかも不用意に開蓋しないロック機構を有する、合成樹脂製のヒンジ付きキャップに係るものである。

[0002]

【従来の技術】従来、ヒンジ付きキャップは、ヒンジ部分を回転軸として蓋が開閉自在となっており、閉蓋時には蓋がキャップ本体の天面を覆った状態で固定され、一方、開蓋時には、蓋の下端面からキャップ本体上端部よりも膨出した突出部に、指を掛けて上方へ引き上げることで係合が解除されて蓋が開けられるものであった。また、不用意に開蓋しないように、キャップ本体と蓋との間にロック機構を設け、開蓋時にはロックを解除して開けるようにしたものがあった。

[0003]

[0004]

【課題を解決するための手段】上記のような問題を解決するため、この発明は、天面に注液口を有し、側面に容器へ係止される係止簡部を有するキャップ本体と、該キャップ本体の天面を覆い、後側に設けられたヒンジを回転軸として開閉可能な蓋とからなり、閉蓋状態ではロックされ、開蓋時にはロックの解除が自在なロック機構を有するヒンジ付きキャップにおいて、蓋の側面部に可撓

性を有し、キャップ本体の天面の両横側及び蓋の両横側 面に係合部を設け、閉蓋時には各々の係合部が係合する ことでロックされ、開蓋時には蓋の前側面を押圧するこ とで蓋の側面が撓み、横側面が広がって係合が解かれる ことでロックが解除されるようにしたものである。

[0005]

【発明の実施の形態】この発明のヒンジ付きキャップは、キャップ本体と蓋とがヒンジで連結され、該ヒンジを回転軸として蓋の開閉がなされる。蓋の側面部は前側面から水平方向の押力で変形するように可撓性を有しており、横側面の内側には対向する位置で係合部が設けられる。キャップ本体の天面の両横側には、蓋の係合部と係合するように、外周に向かって相反する位置で係合部が設けられている。

【0006】ここで、蓋を閉じる場合には、蓋がキャップ本体の天面を覆うように上から押さえ込むことで、蓋の下端部がキャップ本体の天面に設けられた係合部と接触し、蓋の横側面が外周方向に撓んで膨出し、各々の係合部を乗り越えて係合される。このとき、係合部は十分な掛かり量をもって強固に係合されるので、蓋に指を掛けて上方へ引き上げようとしても係合を解除できないロック状態となる。

【0007】次に、蓋を開ける場合には、蓋の前側面を 前方へ押圧することによって前側面が前方へ押し込まれ るように変形し、両側の横側面が外周方向に広がるよう に変形して、キャップ本体と蓋との係合が外されてロッ ク状態が解除される。さらに、前側面を前方へ押圧した まま上方に引き上げることによって、ヒンジを回転軸と して蓋が開けられる。押圧により変形したした蓋の前側 面及び横側面は、指を放すと弾性によって復元し、再び 元の形状に戻る。

[0008]

【実施例】以下、この発明の実施例について図面に基づいて説明する。キャップ本体1と蓋2とはヒンジ3で連結されており、該ヒンジ3を回転軸として蓋2の開閉がなされる。キャップ本体1の側面部には、キャップを容器に固定するための係止胴部11が設けられ、天面4の中央付近には注液口5を有している。一方、蓋2の裏面の中央付近には開栓10を有しており、閉栓10が注液口5を閉塞した状態で、蓋2がキャップ本体1の天面4を覆うように閉じられる。

【0009】蓋2の側面部は上から見て円形状となっており、前側面8から水平方向の押力で変形するように可撓性を有している。また、蓋2の前側面8には、前側面8を押圧したり引き上げたりするときに指が滑らないように突条12が設けられている。横側面9の下端部には蓋2の内側から外側への貫通孔である凹部6が対向する位置で設けられている。キャップ本体1の天面4の両横側には、蓋2の凹部6と係合するように、外周に向かって相反する位置でフック7が設けられている。

【0010】ここで、蓋2を閉じる場合には、蓋2がキャップ本体1の天面4を覆うように上から押さえ込むことで、蓋2の下端部がキャップ本体1の天面4の両機側に設けられたフック7と接触し、蓋2の横側面9が外周方向に押し広げられるように撓んで膨出し、フック7が蓋2の下端部を乗り越えて凹部6に係合される。このとき、フック7と凹部6は十分な掛かり量をもって強固に係合されるので、蓋2の突条12に指を掛けて上方へ引き上げようとしても係合を解除できないロック状態とな

【0011】次に、蓋2を開ける場合には、蓋2の前側面8を前方へ押圧することによって前側面8が前方へ押し込まれるように変形し、両側の横側面9が外周方向に広がるように変形して、蓋2の側面が円形状から楕円形状に変形した状態となり、キャップ本体1のフック7と蓋2の凹部6との係合が外されてロック状態が解除される。さらに、前側面8を前方へ押圧したまま上方に引き上げることによって、ヒンジ3を回転軸として蓋2が開けられる。このとき前側面8に設けられた突条12に指を掛けることにより、指が滑ることなく容易に開蓋することができる。押圧により変形したした蓋2の前側面8及び横側面9は、指を放すと弾性によって復元し、再び元の形状に戻る。

【0012】前側面8を前方に押圧してロック状態を解除するのに必要な押力は、蓋2の側面部の肉厚及び高さによって適宜調整が可能であり、子供には開けられず大人が簡単に開けられる押力として、3Kgf程度が好ましい。また、このヒンジ付きキャップは、合成樹脂を原料として射出成形により一体で簡単に生産することができ、その原料としてはヒンジ性、弾性を有するものとしてたとえばポリプロビレン、ポリエチレンなどの合成樹脂が好ましい。

[0013]

【発明の効果】この発明によれば、閉蓋時の蓋とキャッ

ア本体は、十分な掛かり量をもって強固に係合されたロック状態となっているので、幼児等が手にしたとき、容易に開蓋されて内容物を誤用するという心配がないうえ、このヒンジ付きキャップを何かにぶつけたり引っかけたりしても、不用意に蓋が開いてしまうことがない。また、蓋をキャップ本体の天面へ向けて押さえ込むだけで、閉蓋と同時に蓋を容易にロックすることができるうえ、前側面を前方に押圧しながら上方に引き上げることによって簡単に蓋が開けられるので、ロック機構を解除するための煩わしい操作を必要としない。さらに、このヒンジ付きキャップは、合成樹脂を原料として射出成形により一体で簡単に生産することができるので、簡単な構造で安価に生産できるものである。

【図面の簡単な説明】

【図1】この発明の一実施例を示すヒンジ付きキャップ の開蓋状態を示す外観斜視図である。

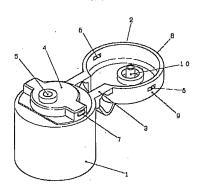
【図2】この実施例のヒンジ付きキャップの閉蓋状態を 示す外観斜視図である。

【図3】この実施例のヒンジ付きキャップの閉蓋状態を 示す縦断面図である。

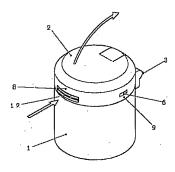
【符号の説明】

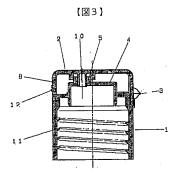
- 1 キャップ本体
- 2 蓋
- 3 ヒンジ
- 4 天面 5 注液口
- 6 凹部
- 7 フック
- 8 前側面
- 9 横側面
- 10 閉栓
- 11 係止筒部
- 12 突条

[図1]



【図2】





① Veröffentlichungsnummer: 0 306 670 B1

EUROPÄISCHE PATENTSCHRIFT

(45) Veröffentlichungstag der Patentschrift: 23.12.92

51 Int. Cl.5: **B65D** 47/06

(21) Anmeldenummer: 88111567.9

(2) Anmeldetag: 19.07.88

- (54) Kunststoffverschluss für einen Kunststoffbehälter.
- (30) Priorität: 09.09.87 DE 3730225 U
- 43 Veröffentlichungstag der Anmeldung: 15.03.89 Patentblatt 89/11
- 45 Bekanntmachung des Hinweises auf die Patenterteilung: 23.12.92 Patentblatt 92/52
- 84) Benannte Vertragsstaaten: DE ES FR GB GR IT NL
- 66 Entgegenhaltungen: FR-A- 2 514 330 US-A- 3 204 827

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Beschreibung

Die Erfindung betrifft einen auf den Hals eines Kunststoffbehälters aufschraubbaren Kunststoffverschluß mit einem Schraubring und mit einem Einsatzteil mit flexiblem Balg und Ausgießtülle, auf deren Außengewinde eine Bügelkappe aufschraubbar ist.

Für das Verschließen von Kunststoffkanistern sind bereits viele Arten von Kunststoffverschlüssen bekannt. Bei der einfachsten Ausführung handelt es sich um eine auf das Außengewinde des Kanisterhalses aufschraubbare Schraubkappe, welche der Benutzer zum Ausgießen abschraubt, um dann direkt über den Kanisterhals das z.B. flüssige Füllgut zu entleeren. Hierbei kann man nur wenig gezielt ausgießen und stellt ein unangenehmes Glucksen während des Ausgießvorganges fest. Dadurch wird teilweise auch Füllgut verschüttet, was je nach Füllgutart unerwünscht ist. Außerdem müssen zur Gewährleistung der Dichtigkeit insbesondere bei kriechfreudigen Produkten stirnseitige Dichtungen zusätzlich aufgelegt werden, durch welche der Preis des Verschlusses für den Hersteller problematisch wird.

Um das Ausgießen zu verbessern, ist es auch bekannt, einen Kunststoffverschluß mit einem flexiblen Balg zu versehen, der von einer Bügelkappe verschlossen werden kann und über eine Schraubkappe auf den Kanisterhals aufschraubbar ist. Diese Ausführungsform ist mit Nachteil dreiteilig und daher für den Hersteller sehr aufwendig. Bei einigen Füllgütern muß zur Gewährleistung einer guten Dichtigkeit des geschlossenen Kanisters wiederum eine zusätzliche Dichtungsscheibe eingelegt werden, wodurch der Aufwand in nachteiliger Weise weiter steigt.

Es ist ferner ein Kunststoffverschluß bekannt, der innenseitig neben den Randsteg eines speziell ausgebildeten Halsrandes eingesetzt und an seinem Umfang mit dem Randsteg verschweißt wird. Dadurch ist der erstmals verschlossene Behälter zwar garantiert flüssigkeitsdicht, die Herstellung ist im allgemeinen aber unerwünscht teuer und nur in speziellen Fällen gerechtfertigt, z.B. wenn Härte und Schmelztemperatur des Kunststoffes des Behälters andere als des Verschlusses sind. Für die üblichen Kunststoffverschlüsse ergibt sich aber aus diesem bekannten Verschluß keine Lösung einer preiswerten Massenherstellung.

Bei anderen Kunststoffverschlüssen mit flexiblem Balg ist anstelle der Verschweißung die Befestigung des Einsatzteiles mit flexiblem Balg und Ausgießtülle über einen übergreifenden und umgelegten Haltering aus Blech erfolgt. Mit diesem läßt sich zwar ein guter Garantieverschluß erreichen, auch die Dichtigkeit ist gewährleistet, der Aufwand des separaten Halteringes auf der Stirnfläche des

Behälterhalses ist aber ebenfalls nur bei besonderen Ausführungsformen zu empfehlen. Eine preiswerte Massenfabrikation von Verschlüssen für normale Kanisterhälse mit Außengewinde in Gestalt einer Schraubkappe kann man aber nach diesem Muster nicht gewährleisten.

Aus diesem Grunde sind Hersteller von Kunststoffverschlüssen bereits dazu übergegangen, die Vielzahl der Verschlußteile zu verringern, insbesondere Schraubkappe und Einsatzteil mit flexiblem Balg in einem Stück zusammenzufassen, so daß nur noch die Bügelkappe separat hergestellt und mit dem Außengewinde der Ausgießtülle verschraubt werden muß. Der Nachteil dieser bekannten Schraubverschlüsse besteht aber in der umständlichen und zeitaufwendigen Produktion des den Balg und die Schraubkappe zusammenfassenden Teils, wobei es außerdem nachteilig ist, daß ein für die Funktion und Faltung des Balges ausreichend weicher Kunststoff zu verwenden ist. Dieser Kunststoff ist andererseits für die Schraubkappe wenig geeignet, die eine gute Festigkeit und Stabilität haben muß, damit sie einerseits einen festen Schraubverschluß gewährleistet und andererseits auch die Anbringung eines Garantieverschlusses ermöglicht. Der Hersteller ist also dazu übergegangen, dieses einstückige Teil im Bereich der Schraubkappe mit dickeren Wandungen zu versehen. Dadurch aber wird die Aushärtzeit und Verweilzeit des Formstückes im Werkzeug unerwünscht lang. Außerdem wird durch die dickere Ausgestaltung der Wandungen zur Erreichung der notwendigen Starrheit und Stabilität die Dichtfunktion nachteilig herabgesetzt mit der Folge, daß wiederum Dichtringe eingelegt werden müssen, um Flüssigkeitskanister durch diesen Verschluß wirklich flüssigkeitsdicht verschließen zu können.

Angesichts dieser Probleme liegt der Erfindung die Aufgabe zugrunde, den Kunststoffverschluß der eingangs genannten Art so weiter zu entwickeln, daß unter Beibehaltung guter Ausgießeigenschaften, z.B. mittels eines flexiblen Balges, die automatische Herstellung vereinfacht und verbilligt wird.

Die Lösung dieser Aufgabe gelingt erfindungsgemäß dadurch, daß die Bügelkappe mit dem Schraubring einstückig ausgebildet ist und die Kopfwand der Bügelkappe in einem Abstand von der Kopfwand des Schraubringes herausstehend angeformt ist. Die Erfindung geht in überraschender Weise einen anderen Weg als der zuletzt beschriebene Verschlußhersteller, denn sie vereinigt nicht das Einsatzteil mit dem flexiblen Balg einerseits mit dem Schraubring andererseits, sondern vereinigt den Schraubring einerseits mit der Bügelkappe andererseits. Dadurch können mit Vorteil die starren und festen Teile des Kunststoffverschlusses aus dem einen Material und mit entsprechend dikken Wandstärken hergestellt werden, während an-

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dererseits das Einsatzteil mit dem flexiblen Balg und ggf. sogar daran angebrachten Dichtteilen aus weicherem Kunststoff und mit ggf. dünneren Wandstärken ausgebildet werden kann.

Man könnte zwar der Auffassung sein, daß zur Vereinfachung der Herstellung eines Verschlusses aus drei Teilen entweder eine erste Zweiergruppe oder eine andere Zweiergruppe mit dem jeweils dritten Teil aus einem Stück hergestellt werden kann. Man könnte also meinen, der Fachmann würde bei Feststellen von Nachteilen der bekannten Verschlüsse ohne weiteres versuchen, Bügelkappe und Schraubring zu einem Stück bei der Herstellung zu vereinigen. Dem stehen aber erhebliche Bedenken entgegen, daß nämlich die Bügel zum Bewegen der Bügelkappe relativ zum Schraubring für den Benutzer zugänglich sein müssen, ohne daß er besondere Werkzeuge einsetzt. Mit anderen Worten muß der Fachmann in der Kopfwand des Schraubringes unbekannte Mittel vorsehen, um die in dieser Ebene liegenden Bügel der Bügelkappe ergreifen zu können. Solche Mittel, z.B. Ausnehmungen oder Greifhilfen, sind aber kaum denkbar.

Hingegen ist zur Überwindung dieser Bedenken die Erfindung den Weg gegangen, zwischen der Kopfwand des Schraubringes einerseits und der Kopfwand der Bügelkappe andererseits einen Abstand derart vorzusehen, daß aus dem Bereich des (größeren) Schraubringes (innen) die Bügelkappe ein Stück weit (Abstand) heraussteht. Gerade hierdurch kann in überraschend einfacher Weise erreicht werden, daß die Bügel, die nur an bestimmten Anlenkstellen mit der Kopfwand der Bügelkappe verbunden sind, frei liegen und für den Benutzer zum Öffnen ohne den Gebrauch von Werkzeug zugänglich sind.

Durch die vorstehend erläuterten Maßnahmen gemäß der Erfindung wird also der Einsatzteil mit dem flexiblen Balg zur Beibehaltung der bekannten guten Ausgießeigenschaften bei dem neuen Kunststoffverschluß aufrechterhalten, und die Herstellung mittels wenigstens teilweiser automatischer Maschinen kann vereinfacht und verbilligt werden. Bei Kunststoffverschlüssen handelt es sich in aller Regel um Massenprodukte, so daß schon kleinere Vereinfachungsschritte bei der automatischen Herstellung große Wirkungen haben.

Es versteht sich, daß der Benutzer mit der Bügelkappe den flexiblen Balg zum Öffnen aus dem Bereich innerhalb des Schraubringes herauszieht und dann aus dem Einsatzteil mit der Ausgießtülle abschraubt, so daß der Benutzer dann den Griff zum Herausreißen des Garantiesiegels erfassen und damit die Packung auf übliche Weise korrekt öffnen kann.

Der neue Verschluß gemäß der Erfindung erlaubt auch einen Garantieverschluß, denn es ist bekannt, daß man einfache Schraubringe außen unten neben dem Ansatzbereich zwischen Hals und Kanister mit einem Garantieband versehen kann. Weil mit Vorteil der Schraubring aus hartem Kunststoff und mit ggf. dickeren Wandungen hergestellt werden kann, hat er eine ausreichende Steifigkeit, so daß auch unerwünschte Manipulationen nicht ohne sichtbare Beschädigung bleiben bzw. so ausgeschlossen sind, daß der mit dem neuen Verschluß versehene Behälter sichere Verschlußgarantien gewährleistet.

Die Erfindung ist vorteilhaft weiter dadurch ausgestaltet, daß die Bügelkappe über eine Reißhaut mit dem Schraubring verbunden ist. Bildet man nämlich Schraubring und Bügelkappe als einstückiges Formteil aus, dann ergeben sich für den gewöhnlichen Herstellungsfachmann, der eine solche Vereinigung von zwei Teilen zu einem einzigen Spritzteil in Erwägung zieht, die Bedenken, wie die Bügelkappe gegenüber dem Schraubring bewegt werden kann, wenn doch beide aus einem einzigen Stück bestehen. Ordnet man zwischen dem einen und dem anderen Stück an definierten Bereichen eine Sollbruchstelle, z.B. in Gestalt einer Reißhaut, aus, dann ergibt sich für den Benutzer von selbst, daß er die Bügelkappe zum Herausreißen des flexiblen Balges beim Öffnen des Verschlusses ohne großen Kraftaufwand aufreißen kann, wodurch dann die Bügelkappe separiert ist und gegenüber dem Schraubring gedreht werden kann.

Die Anordnung der Kopfwand der Bügelkappe in der einen äußeren Ebene und die Anordnung der Kopfwand des Schraubringes andererseits in der anderen inneren Ebene, im Abstand der beiden Ebenen voneinander, gelingt besonders zweckmäßig dann, wenn erfindungsgemäß die Kopfwand des Schraubringes mit wenigstens einem erhaben nach außen vorstehenden Haltestück einer Höhe etwa gleich dem Abstand zur Kopfwand der Bügelkappe versehen ist. Hier kann zwar die Anbringung eines Haltestückes am Schraubring genügen, zweckmäßig wird aber die Anbringung wenigstens zweier Haltestücke sein, durch welche ein ausreichend stabiles Kunststoffteil gebildet ist, bei dem der Benutzer sofort die frei zugänglichen Bügel erkennt, die er beim Öffnen betätigt.

Besonders zweckmäßig ist es erfindungsgemäß, wenn das Haltestück in Draufsicht die Form eines Kreisringabschnittes hat. Hier kann man sich vorstellen, daß der Schraubring selbst aus der zylindermantelförmigen Fläche besteht, auf der innen zum Aufschrauben ein Gewinde und außen ggf. eine Riffelung angeordnet sind, wobei nach innen in Richtung Behältermitte eine freie Öffnung verbleibt und nach oben außen sich an den Umfang die ringförmige Kopfwand anschließt, die in der Mitte ebenfalls eine Kreisöffnung frei läßt. Längs eines Teils dieses Kreises ist nun das Haltestück angeformt zu denken, welches einen ähnlichen

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Querschnitt wie der Schraubring selbst hat, wenn auch mit anderen Maßen und Proportionen. In Draufsicht iedenfalls kann das Haltestück zweckmäßig wie ein Kreisringabschnitt ausgebildet sein. Im Querschnitt ergibt sich wiederum L-Gestalt, d.h. an den Teilzylindermantel schließt sich ein Kreisringabschnitt an, welcher in der gleichen Ebene wie die Kopfwand der Bügelkappe liegt. Dabei ist es zweckmäßig, wenn der Übergang von diesem Kreisringabschnitt zur Kopfwand der Bügelkappe über die Reißhaut erfolgt. Die Erfindung schlägt daher vor, daß zwei sich diametral gegenüberliegende, bogenförmige Haltestücke am Schraubring angeformt sind und die Reißhaut, dem Bogen der Haltestücke folgend, sich von den Haltestücken radial nach einwärts erstreckt. Wenn sich also zwei bogenförmige Haltestücke einander gegenüberliegen, gibt es auch zwei entsprechend bogenförmige Reißhäute, so daß sich die Kopfwand der Bügelkappe dazwischen befindet. Die Praxis hat gezeigt, daß nach Hochbiegen der ebenfalls im Bereich der Haltestücke angelenkten Bügel ein Herausreißen und Zerstören der Reißhäute beim Einleiten des Öffnungsvorganges, d.h. Herausziehen des flexiblen Balges mittels der Bügel, unschwer möglich

Zweckmäßig ist es gemäß der Erfindung ferner, wenn am Einsatzteil auf der der Ausgießtülle gegenüberliegenden Seite des flexiblen Balges ein Dichtflansch angeformt ist. Es war eingangs verschiedentlich darauf hingewiesen worden, daß bei kriechfähigen Füllgütern die Dichtung oft ein Problem ist. Zur Vermeidung separater Dichtscheiben oder Gummiringe, die in einem gesonderten Arbeitsgang zusätzlich eingelegt werden müßten, schlägt die Erfindung vor, das Einsatzteil aus hinreichend weichem und elastischem Kunststoff mit geeignet dünnen Dichtflanschen auszubilden, so daß sich das Einlegen einer zusätzlichen Dichtung mit Vorteil erübrigt. Der Dichtflansch ist bei dem hier vorgeschlagenen Verschluß in günstiger Weise ringförmig und kann sich stirnseitig auf die Ringfläche des Behälterhalses legen und dort den Schraubring festklemmen lassen.

Wenn eine solche Dichtung aber noch nicht ausreichen sollte, kann man gemäß weiterer vorteilhafter Ausgestaltung der Erfindung einen Dichtkonus am Dichtflansch zusätzlich anbringen. Während sich der Dichtflansch parallel zur Ebene der Kopfwände erstreckt, liegt die Erstreckung des Dichtkonus senkrecht dazu, nämlich in Achsrichtung des Behälterhalses. Dieser Dichtkonus ist ebenfalls ringförmig und verläuft in einem solchen Abstand vom äußeren Ende des Dichtflansches, daß er in gute Klemmlage auf die Innenseite des Behälterhalses zu liegen kommt.

Die Stirnfläche des Behälterhalses kann bei der Herstellung bisweilen verkratzt oder anderweitig beschädigt sein, oder es können Einfallstellen vorliegen. Selbst wenn also der Dichtflansch am Einsatzteil für sich eine gute Dichtigkeit gewährleisten würde, könnten doch kleinste Leckagen auftreten, die dann aber ausgeschaltet sind, wenn zusätzlich der Dichtkonus vorgesehen ist. Man hat durch den Dichtkonus praktisch eine doppelte Dichtung im Bereich des ringförmigen oberen, äußeren Endes des Behälterhalses.

Eine weitere Verbesserung der Dichtmöglichkeit am äußeren Ende des Behälterhalses besteht darin, wenn erfindungsgemäß auf der dem Behälterhals zugewandten Fläche des Dichtflansches außerdem ein Ringvorsprung angebracht ist. Dieser kann sich beispielsweise in der Mitte zwischen der kreisförmigen Außenkante des Dichtflansches und der Ansatzkante neben dem Dichtkonus befinden. Im Querschnitt gesehen kann dieser Ringsvorsprung dreieckförmig, kreisförmig, wulstförmig, vieleckig oder dergleichen ausgestaltet sein. Das Anformen eines Ringvorsprunges an den Dichtflansch erfolgt mit einfachen Mitteln in ein und derselben Spritzmaschine beim Formen des Einsatzteiles.

Durch den Verschluß gemäß der Erfindung kann also nicht nur unter Beibehaltung guter Ausgießeigenschaften, insbesondere mittels des flexiblen Balges, die automatische Herstellung vereinfacht und verbilligt werden, indem beispielsweise zwei der drei Teile, nämlich der Schraubring und die Bügelkappe, zu einem Teil vereinigt werden, sondern es kann gleichzeitig auch eine einwandfreie Dichtigkeit erreicht werden, weil der Einsatzteil aus weicherem Kunststoff mit dünneren Wandungen ausgestaltet wird und an den entsprechenden Stellen mit Dichtmitteln (Dichtflansch, Dichtkonus, Gewinde etc.) versehen werden kann.

Ein solcher Verschluß ist auf die üblichen Behälterhälse aufschraubbar und gewährleistet eine Gerantiesicherheit, weil Garantiebänder oder ähnl. ohne weiteres angebracht sein können.

Weitere Vorteile, Merkmale und Anwendungsmöglichkeiten der vorliegenden Erfindung ergeben sich aus der folgenden Beschreibung bevorzugter Ausführungsbeispiele in Verbindung mit den anliegenden Zeichnungen. Es zeigen:

- Fig. 1 Abgebrochen die Querschnittsansicht eines Kunststoffverschlusses gemäß der Erfindung mit Behälterhals im geschlossenen Zustand als Schnitt entlang der Linie B-B der Fig. 3,
- Fig. 2 eine ähnliche Darstellung wie Fig. 1, wobei hier jedoch der Querschnitt entlang der Linie A-A der Fig. 3 genommen ist,
- Fig. 3 eine Draufsicht auf den Kunststoffverschluß mit einigen gestrichelt gezeigten Innenteilen und

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Fig. 4 eine vergrößerte und abgeschnittene Querschnittsansicht gemäß dem strichpunktierten Kreis IV in Fig. 1 unter Weglassung von Schraubring und Behälterhals.

Die in den Zeichnungen veranschaulichten Ausführungsformen zeigen einen Kunststoffverschluß zum Verschließen eines Kunststoffkanisters, in dem sich beispielsweise Motoröl befindet.

Der allgemein mit 1 bezeichnete Hals des Kunststoffkanisters weist im Bereich 2 eine Verzahnung auf für den Eingriff mit Sperrlaschen 3 zur Fixierung des Garantiebandes 4 an dem allgemein mit 5 bezeichneten Schraubring, der aus der zylindermantelförmigen Wandung 6 mit Außenriffelung 7 einerseits und Kopfband 8 andererseits besteht. Das Garantieband 4 des Schraubringes 5 ist unten an diesem angebracht, d.h. auf der Seite zum Kanister hin, bzw. von außerhalb gesehen in Richtung des Inneren des Kanisters.

Außen, d.h. in der Darstellung der Fig. 1 und 2 oben bzw. auf der dem Kanisterinneren am weitesten entfernt gelegenen Seite sind zwei diametral gegenüberliegende, bogenförmige Haltestücke 9 angeformt.

Der Kunststoffverschluß besteht im wesentlichen aus zwei Teilen, nämlich dem bereits beschriebenen Schraubring 5 aus einem härteren Kunststoff mit dicken Wandungen für eine ausreichende Stabilität des Innengewindes 10; und aus dem zweiten, flexiblen Teil, vorzugsweise aus Polyvenylacetat, wobei dieses allgemein mit Einsatzteil 11 bezeichnet ist.

Das Einsatzteil 11 umfaßt seinerseits den Dichtflansch 12, den daran angeformten ringförmigen Dichtkonus 13, die beide zusammen im Querschnitt L-Form haben, wie man aus den Fig. 1, 2 und 4 erkennt, ferner den von der Verbindung der beiden Schenkel dieses L ausgehenden und daran angeformten flexiblen Balg 14 und das äußere Teil unter Bildung der Ausgießtülle 15, auf deren Innenseite das Garantiesiegel 16 mit Greifband 17 über eine kreisförmige Sollbruchlinie 18 angeformt ist und die auf der Außenseite mit dem Außengewinde 19 versehen ist.

Auf dieses Außengewinde 19 ist mit entsprechendem Innengewinde 20 die allgemein mit 21 bezeichnete Bügelkappe aufgeschraubt, die einstückig mit dem Schraubring 5 ausgeformt ist.

Einstückig werden Schraubring 5 und Bügelkappe 21 dadurch, daß sie längs zweier bogenförmiger, diametral einander gegegenüberliegender Reißhäute 22 miteinander verbunden sind. Die jeweilige Reißhaut schließt sich direkt an die bogenförmigen Haltestücke 9 an, und zwar nahe der Oberfläche der Kopwand 23 der Bügelkappe 21. Aus der Darstellung der Fig. 1, in welcher die Reißhaut 22 im Querschnitt wie eine Sollbruchlinie

verdünnt dargestellt ist, sieht man, wie das Haltestück 9 im Querschnitt ebenfalls L-förmig derart ist, daß anschließend an die Kopfwand 8 des Schraubringes 5 ein zylindermantelförmiger Teil nach "oben außen" hochragt und von dort der andere Schenkel angesetzt ist, der sich etwa in der Ebene der Kopfwand 23 der Bügelkappe 21 befindet und in der genannten Reißhaut 22 endet.

In Richtung auf den Mittelpunkt M (Fig. 3) der Bügelkappe 21 hin ist neben der Reißhaut 22 das Material des Bügels 24 der Bügelkappe 21 gezeigt. Beide in Fig. 3 von oben vollständig sichtbare Bügel 24 sind spiegelbildlich verkehrt gleich ausgestaltet und im Bereich der Achse a-a in der Kopwand 23 der Bügelkappe 21, welche auch durch den Mittelpunkt M läuft, im Bereich 25 angelenkt. Bei der Darstellung der Fig. 2 längs der Linie A-A der Fig. 3 erkennt man deutlich, daß der Bügel 24 ohne direkte Anbindung (in diesem Längsschnittbereich) an den Schraubring 5 frei nach außen ragt. Dagegen ist der Bügel 24 im Bereich der bogenförmigen Haltestützen (wie Fig. 1 im Schnitt zeigt) über die Reißhaut 22 verbunden.

Der Benutzer kann also im Bereich der Haltestützen die Bügel von außen nicht ohne weiteres ergreifen, weshalb im Bereich der Haltestützen, gemäß Darstellung der Fig. 3 in deren Mitte, der Anlenkbereich 25 vorgesehen ist. Das Ergreifen der Bügel ist aber in beiden Lagen um 90° zum Anlenkbereich 25 versetzt gut möglich, weil gemäß Darstellung der Fig. 2 die Bügel 24 hier zum Ergreifen frei liegen.

Die wichtige Neuerung an der in den Fig. 1 bis 3 dargestellten Ausführungsform des Kunststoffverschlusses liegt also in der zweiteiligen Ausführungsform, wobei das eine Teil das Einsatzteil 11 ist und das andere Teil der Schraubring 5 ist, an welchem oben über die beiden bogenförmigen Reißhäute 22 auch die Bügelkappe 21 im Zuge desselben Herstellungsprozesses und aus demselben Kunststoffmaterial angeformt ist. Man könnte den Schraubring 5 und die Bügelkappe 21 insgesamt als "Schraubkappe" bezeichnen.

Die bogenförmigen Haltestücke 9 haben in der Draufsicht der Fig. 3 die Form eine Kreisringabschnittes. Im Querschnitt haben sie die in Fig. 1 dargestellte L-Form. Der in Richtung der Achse der Ausgießtülle 15 verlaufende Steg 9 hat dabei eine Höhe d, durch welche die untere Fläche der Kopfwand 23 der Bügelkappe 21 im Abstand von der Unterfläche der Kopfwand 8 des Schraubringes 5 gehalten wird. Aus diesem Grunde erscheint der Abstand d auch in Fig. 2.

Wäre dieser Abstand d gleich Null, d.h. würde die Kopfwand 23 der Bügelkappe 21 in der gleichen Ebene wie die Kopfwand 8 des Schraubringes 5 liegen, dann hätte der Benutzer keine Möglichkeit, ohne Anwendung von Werkzeugen die Bü-

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gel 21 zu ergreifen. Außer diesem ersichtlichen Nachteil ergäbe sich der weitere Nachteil, daß beim automatischen Erfassen des Schraubringes 5 und Aufschrauben auf den Behälterhals 1 radial auftretende Kräfte direkt auf die Reißhaut 22 übertragen würden mit der Folge, daß schon bei der Montage des Kunststoffverschlusses die Reißhaut 22 ganz oder teilweise beschädigt wäre. Durch den nach außen hochstehenden Schenkel des L-förmigen Haltestückes 9 wird hingegen an der Anlenkstelle dieses Schenkels oder Haltestückes 9 an der radial inneren Kante der Kopfwand 8 des Schraubringes 5 ein Gelenk geschaffen, welches beim Betrieb die unvermeidlichen radial wirkenden Kräfte des automatischen Werkzeuges beim Ergreifen des Schraubringes aufnimmt unter Veränderung der Lage der horizontalen Kopfwand 8 und des vertikalen Zylindermantelabschnittes des Haltestückes 9 bzw. unter Veränderung des Winkels zwischen diesen beiden normalerweise etwa senkrecht aufeinanderstehenden Wandungen. Eine Beschädigung oder gar ein Aufreißen der Reißhaut 22 bei der Montage ist also mit Vorteil ausgeschaltet.

Aus den Fig. 1, 2 und 4 sieht man ferner, daß am Einsatzteil 11 auf der der Ausgießtülle 15 gegenüberliegenden Seite des flexiblen Balges 14 ein Dichtflansch 12 angeformt ist. Dieser kann aus dem gleichen flexiblen und weichen Kunststoffmaterial hergestellt sein wie der flexible Balg 14. Der Dichtflansch 12 ist also ebenso flexibel und elastisch nachgiebig. Wird der Dichtflansch 12 in der in den Fig. 1 und 2 gezeigten Weise zwischen die stirnseitige Fläche des Behälterhalses 1 einerseits und die untere oder innere Fläche der Kopfwand 8 des Schraubringes 5 andererseits eingelegt und durch Festschrauben des Schraubringes über den Eingriff des Gewindes 10 zusammengedrückt, dann ergibt sich eine zuverlässige Dichtung, die bei der dargestellten Ausführungsform Kreisringform hat.

Sollte es bei der Herstellung zu Beschädigungen der stirnseitigen Ringfläche des Behälterhalses 1 kommen, so daß über den Dichtflansch 12 nicht eine stets einwandfreie Dichtung gewährleistet ist, dann wird die endgültige Dichtfunktion durch einen Dichtkonus 13 erreicht, der aus dem gleichen Material hergestellt ist wie der Dichtflansch 12 und der flexible Balg 14. Der Dichtkonus 13 ist ebenfalls ringförmig und einstückig am Dichtflansch einerseits und Balg 14 andererseits angeformt, und zwar an der Verbindungsstelle dieser beiden Teile miteinander. Während der Dichtflansch 12 in etwa horizontaler Ebene angeordnet ist, d.h. in der Ebene des stirnseitigen Ringes des Behälterhalses 1, erstreckt sich die Wandung des Dichtkonus 13 senkrecht dazu und verläuft damit wenigstens mit einer Oberfläche parallel zur inneren hülsenförmigen Fläche des Behälterhalses 1. Der Dichtkonus 13 liegt unter satter Berührung auf dieser Oberfläche des Behälterhalses von innen auf, so daß durch den Dichtflansch 12 und den Dichtkonus 13 eine doppelte Dichtfunktion erreicht ist.

Fig. 4 zeigt als Alternative zu der Darstellung der Fig. 1 und 2 einen Dichtflansch 12, der zusätzlich einen Ringvorvorsprung 26 hat. Dieser legt sich bei der Montage gemäß Fig. 1 und 2 auf die stirnseitige Ringfläche des Behälterhalses 1 und schafft zuverlässig eine ringförmige Dichtung. Dabei ist es auch möglich, daß das in Fig. 4 gezeigte dreieckförmige Profil dieses Ringvorsprunges 26 eine andere Gestalt hat, z.B. Kreisform, oval, vieleckig oder dergl.

Im Betrieb ist zunächst die Herstellung eines solchen Kunststoffverschlusses einfacher. Es wird aus dem ersten Material ein erstes Teil, bestehend aus Schraubring 5 und Bügelkappe 21 geformt. Ferner wird aus einem zweiten weicheren Material (beides Kunststoff) das Einsatzteil 11 mit Dichtflansch 12, Dichtkonus 13, flexiblem Balg 14 und Ausgießtülle 15 geformt (es versteht sich dabei, daß das Garantiesiegel 16 mit Reißlinie 18 und Greifband 17 angeformt ist).

Bei der Montage wird nun der Schraubring 5 samt Bügelkappe 21 ergriffen und nach Aufsetzen des Einsatzteils 11 auf den Hals 1 des Behälters so aufgebracht, daß die zylindermantelförmige Wandung 27 der Bügelkappe 21 mit dem Innengewinde 20 auf das Außengewinde 19 der Ausgießtülle 15 zum einen; das Innengewinde 10 der zylindermantelförmigen Wandung 6 der Schraubkappe 5 in Eingriff mit dem Außengewinde 28 des Behälterhalses 1 zum zweiten in Eingriff gebracht wird; und die Sperrlaschen 3 mit der Verzahnung 2 des Halses 1 zum dritten in Garantieverschlußposition gebracht werden. Danach ist der Behälter verschlossen und der Garantieverschluß angebracht.

Der Benutzer ergreift nun ohne die Notwendigkeit von Werkzeugen die Bügel 24, reißt sie zum Öffnen längs der als Schwächungslinie oder Sollbruchlinie ausgestalteten Reißhaut 22 hoch, bis die Bruchlinie der Reißhaut 22 an den Anlenkbereich 25 kommt. Jetzt zieht der Benutzer beide hochgestellten Bügel 24 weiter nach oben und außen in Richtung der Achse der Ausgießtülle 15 und vom Kanistermittelpunkt fort. Dadurch reißt die Reißhaut 22 auch im Anlenkbereich 25 ab, so daß die Bügelkappe 21 vom Schraubring 5 separiert ist. Danach kann der Benutzer das Mittelteil, bestehend aus flexiblem Balg 14 und Ausgießtülle 15 mitsamt Bügelkappe 21 aus dem Schraubring und Behälterhals 1 nach oben herausziehen. Dabei wird der flexible Balg 14 in bekannter Weise verformt.

Der Benutzer kann nun die Bügelkappe 21 abschrauben, wobei die Gewinde 19 und 20 außer Eingriff kommen. Der Benutzer ergreift nun das Greifband 17 und reißt das Garantiesiegel 16 längs der Kreisbruchlinie 18 heraus. Der Behälter ist ge-

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öffnet, das Füllgut kann nun über die lange Ausgießtülle in gezieltem Strahl ausgegossen werden.

Patentansprüche

- 1. Auf den Hals (1) eines Kunststoffbehälters aufschraubbarer Kunststoffverschluß mit einem Schraubring (5) und mit einem Einsatzteil (11) mit flexiblem Balg (14) und Ausgießtülle (15), auf deren Außengewinde (19) eine Bügelkappe (21) aufschraubbar ist, dadurch gekennzeichnet, daß die Bügelkappe (21) mit dem Schraubring (5) einstückig ausgebildet ist und die Kopfwand (23) der Bügelkappe (21) in einem Abstand (d) von der Kopfwand (8) des Schraubringes (5) herausstehend angeformt ist
- Kunststoffverschluß nach Anspruch 1, dadurch gekennzeichnet, daß die Bügelkappe (21) über eine Reißhaut (22) mit dem Schraubring (5) verbunden ist.
- 3. Kunststoffverschluß nach Anspruch 1 oder 2, dadurch gekennzeichnet, daß die Kopfwand (8) des Schraubringes (5) mit wenigstens einem erhaben nach außen vorstehenden Haltestück (9) einer Höhe etwa gleich dem Abstand (d) zur Kopwand (23) der Bügelkappe (21) versehen ist.
- 4. Kunststoffverschluß nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß das Haltestück (9) in Draufsicht die Form eines Kreisringabschnittes hat.
- Kunststoffverschluß nach einem der Ansprüche 1 bis 4, dadurch gekennzeichnet, daß zwei sich diametral gegenüberliegende, bogenförmige Haltestücke (9) am Schraubring (5) angeformt sind und die Reißhaut (22), dem Bogen der Haltestücke (9) folgend, sich von den Haltestücken (9) radial nach einwärts erstreckt.
- 6. Kunststoffverschluß nach einem der Ansprüche 1 bis 5, dadurch gekennzeichnet, daß am Einsatzteil (11) auf der der Ausgießtülle (15) gegenüberliegenden Seite des flexiblen Balges (14) ein Dichtflansch (12) angeformt ist.
- Kunststoffverschluß nach einem der Ansprüche
 bis 6, dadurch gekennzeichnet, daß ein Dichtkonus (13) am Dichtflansch (12) angebracht ist.
- 8. Kunststoffverschluß nach einem der Ansprüche 1 bis 7, dadurch gekennzeichnet, daß auf der dem Behälterhals (1) zugewandten Fläche des

Dichtflansches (12) ein Ringvorsprung (26) angebracht ist (Fig. 4).

Claims

- 1. Plastic closure which can be screwed onto the neck (1) of a plastic container and having a screw ring (5) and an insert part (11) with a flexible diaphragm (14) and pouring spout (15), onto whose external thread (19) can be screwed a clip cap (21), characterized in that the clip cap (21) is constructed in one piece with the screw ring and the end wall (23) of the clip cap (21) is shaped so as to project by a distance (d) from the end wall (8) of the screw ring (5).
- 2. Plastic closure according to claim 1, characterized in that the clip cap (21) is connected by means of a tearing membrane (22) to the screw ring (5).
- 3. Plastic closure according to claims 1 or 2, characterized in that the end wall (8) of the screw ring (5) is provided with at least one outwardly projecting holding piece (9) with a height roughly the same as the distance (d) from the end wall (23) of the clip cap (21).
- 4. Plastic closure according to one of the claims 1 to 3, characterized in that, in plan view, the holding piece (9) is shaped like a circular ring portion.
- 5. Plastic closure according to one of the claims 1 to 4, characterized in that two diametrically facing, curved holding pieces (9) are shaped onto the screw ring (5) and the tearing membrane (22), following the arc of the holding pieces (9), extends radially inwards from said holding pieces (9).
 - 6. Plastic closure according to one of the claims 1 to 5, characterized in that a sealing flange (12) is shaped onto the insert part (11) on the side of the flexible diaphragm (14) facing the pouring spout (15).
 - Plastic closure according to one of the claims
 to 6, characterized in that a sealing cone (13) is fitted to the sealing flange (12).
 - 8. Plastic closure according to one of the claims 1 to 7, characterized in that a ring projection (26) is fitted to the surface of the sealing flange (12) facing the container neck (1). (fig. 4).

Revendications

- 1. Fermeture plastique pouvant être vissée sur le goulot (1) d'un récipient en plastique, comportant une bague filetée (5) et une pièce d'insertion (11) avec un soufflet souple (14) et un bec verseur (15),sur le filetage extérieur (19) de laquelle peut être vissée une capsule à étrier (21), caractérisée en ce que la capsule à étrier (21) est conçue d'une manière monobloc avec la bague filetée (5), et que la paroi de face (23) de la capsule à étrier (21) est rapportée en saillie, à une distance (d) de la paroi de face (8) de la bague filetée (5).
- 2. Fermeture plastique selon la revendication 1, caractérisée en ce que la capsule à étrier (21) est liée à la bague filetée (5) par l'intermédiaire d'une pellicule arrachable (22).
- 3. Fermeture plastique selon la revendication 1 ou 2, caractérisée en ce que la paroi de face (8) de la bague filetée (5) est pourvue d'au moins un élément de soutien (9), en saillie en relief vers l'extérieur, ayant une hauteur approximativement égale à la distance (d) à la paroi de face (23) de la capsule à étrier (21).
- 4. Fermeture plastique selon l'une des revendications 1 à 3, caractérisée en ce que l'élément de soutien (9) présente en vue de dessus la forme d'un segment de cercle.
- 5. Fermeture plastique selon l'une des revendications 1 à 4, caractérisée en ce que deux éléments de soutien en forme d'arc (9), diamétralement opposés, sont rapportés à la bague filetée (5), et que la pellicule arrachable (22), qui suit l'arc des éléments de soutien (9), s'étend radialement vers l'intérieur à partir des éléments de soutien (9).
- 6. Fermeture plastique selon l'une des revendications 1 à 5, caractérisée en ce qu'une collerette d'étanchéité (12) est rapportée à la pièce d'insertion (11) sur le côté du soufflet souple (14) opposé au bec verseur (15).
- 7. Fermeture plastique selon l'une des revendications 1 à 6, caractérisée en ce qu'un cône d'étanchéité (13) est rapporté à la collerette d'étanchéité (12).
- 8. Fermeture plastique selon l'une des revendications 1 à 7, caractérisée en ce qu'une saillie annulaire (26) est rapportée sur la surface de la collerette d'étanchéité (12) dirigée vers le goulot (1) du récipient (Figure 4).

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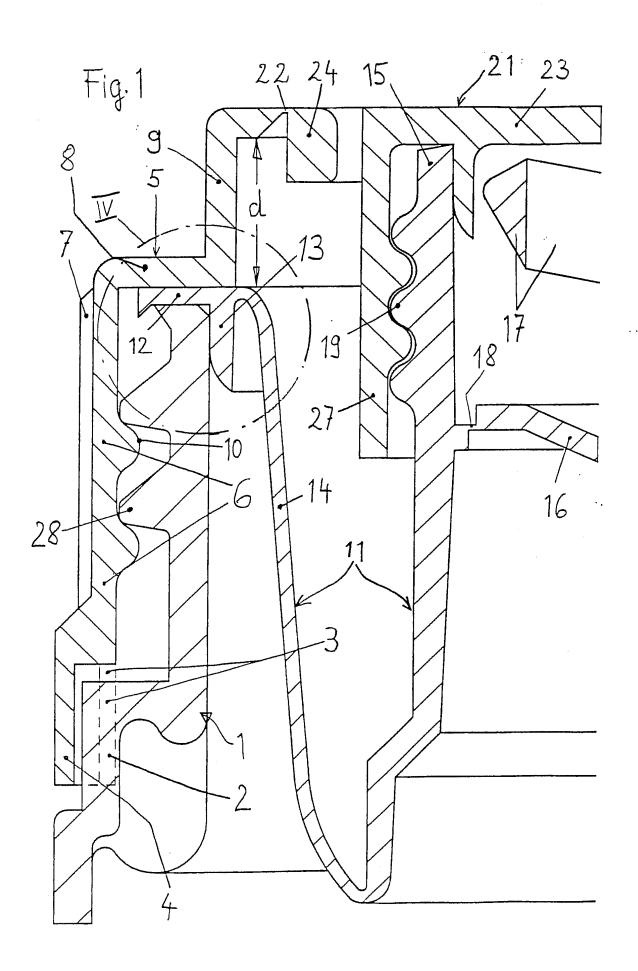
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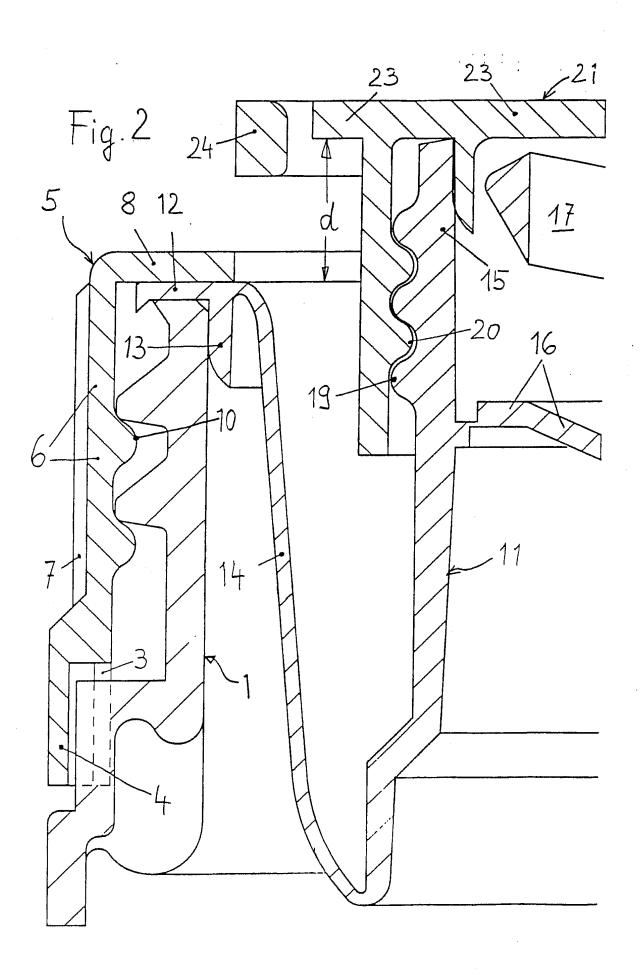
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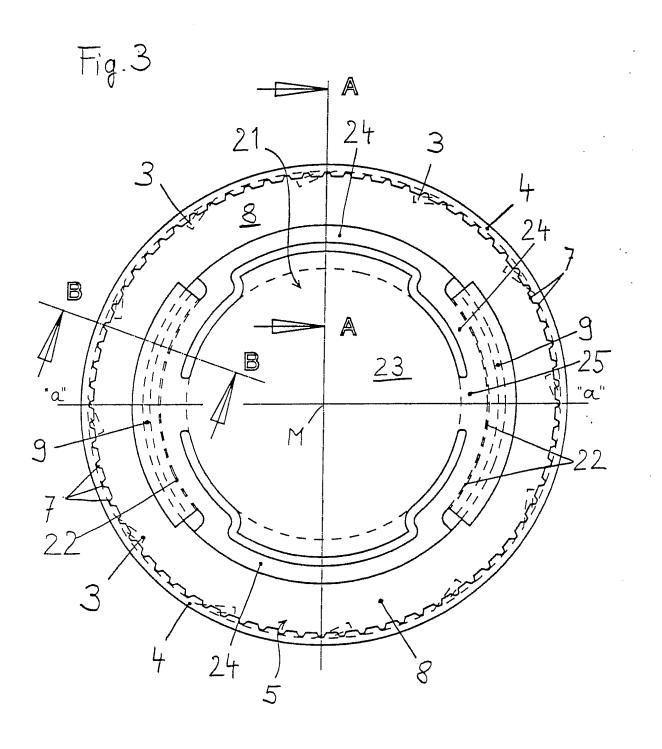
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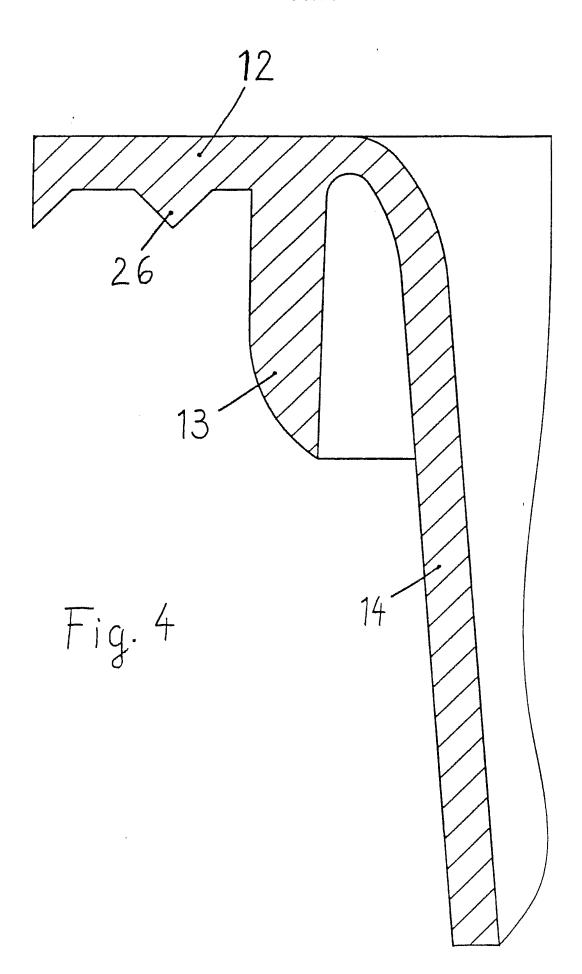
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11 Publication number:

0 236 136 B1

(12)

EUROPEAN PATENT SPECIFICATION

(45) Date of publication of patent specification: 26.05.93 (51) Int. CI.5. C11D 17/04

(21) Application number: 87301905.3

22 Date of filing: 05.03.87

Product for dispensing treatment agents in a washing or dishwashing machine.

30 Priority: 07.03.86 GB 8605734

Date of publication of application:09.09.87 Bulletin 87/37

Publication of the grant of the patent:26.05.93 Bulletin 93/21

Designated Contracting States:

AT BE CH DE ES FR GB GR IT LI NL SE

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Description

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The present invention relates to a product in the form of a multicompartment sachet for the delivery of treatment agents, for example, detergent, bleach, enzyme, rinse conditioner or rinse aid, into the wash liquor in an automatic washing machine or dishwasher.

Multicompartment sachets for delivering ingredients in washing machines in a sequential manner have been disclosed in the prior art.

GB 2 000 177B (Akzo) discloses a two-compartment sachet containing a phosphate free detergent composition based on sodium carbonate. A first compartment is bounded by a water-permeable wall and separated by a partition from a second compartment bounded by an impermeable wall; the partition wall is of material that disintegrates in the wash water. The contents of the second compartment are therefore not released until the contents of the first compartment have been leached out and the partition wall has disintegrated.

US 4 410 441 (Davies et al/Lever Brothers Company) discloses another two-compartment sachet which differs from that of GB 2 000 177B in that the partition wall is of water-insoluble water-permeable material. Release of the contents of the second compartment is by leaching out through the porous partition after the contents of the first compartment have been delivered.

EP 143 476A (Akzo) discloses a sachet having two compartments, one being formed of water – permeable material or material that immediately disintegrates in water and the other being formed of water – impermeable non – disintegrating material and being sealed with a water – sensitive composition comprising an anionic and/or nonionic water – binding polymer, for example, polyvinyl pyrrolidone and a cationic polymeric adhesive, for example, polyethyleneimine. The first compartment releases its contents rapidly, while release from the second compartment is delayed by the slow opening of the water – sensitive seals.

EP 66 463B (Unilever) discloses an article for releasing an active material in a controlled manner, comprising two layers of sheet material (laminates each consisting of an outer porous layer and an inner plastics film layer) bonded together in a grid pattern to form an array of cells or compartments. The sheet material is pinholed for release of active material. If desired different compartments can contain different active material and can be pinholed to differing extents to allow release of different active material at different rates.

EP-A-11502 (Unilever) describes sachets for detergent products in which the sachet material carries a water-soluble or otherwise removable layer coated on or impregnated into the bag material. This document also mentions the possibility of providing a plurality of sachet compartments in order to separate ingredients of the overall composition.

The present invention provides a product for the delivery of treatment agents into the wash liquor of an automatic washing or dishwashing machine, comprising a sachet having at least two compartments, including:

- (i) a first compartment of water insoluble material containing a first treatment agent, said first compart ment having at least one opening zeal and/or being formed of porous water permeable material, and being capable of releasing said first treatment agent into the wash liquor of a washing or dishwashing machine within a period of 3 minutes from the start of a wash process carried out with wash water at 30°C; and
- (ii) a second, non-opening, compartment containing a second, water-soluble or water-dispersible, treatment agent in particulate form, said second compartment being formed at least partially of porous water-permeable material through which said second treatment agent can be leached out by the wash liquor, said second compartment being provided with means for retarding release from the second compartment so as to take place more gradually than release from the first compartment, and/or delaying release from the second compartment for at least 5 minutes from the start of a wash process carried out with wash water at 40 °C, said means for retarding or delaying release comprising
 - (a) a substantially wholly pore-occluding external coating or layer that is capable of being disrupted by the wash liquor, and/or
 - (b) the enclosure of the second compartment within another sachet compartment of porous water permeable material.

The sachet of the invention contains at least two different compartments and is designed to deliver their contents in distinctly different ways: the contents of the first compartment are released very rapidly while the release of the contents of the second compartment is delayed and/or retarded.

In a preferred embodiment of the invention, the sachet is designed to deliver its contents in at least two discrete stages, with an interval between the deliveries of at least 2 minutes, preferably at least 5 minutes

and advantageously from 10 to 15 minutes, from at least two different compartments. The contents of the compartments may if desired be identical, but the sachet of the invention is especially useful for delivering different ingredients at different stages in the washing or dishwashing process.

The contents of the second compartment are in pulverulent or granular form while the contents of the first compartment can be in any desired form compatible with the nature of the compartment's walls and seals. Any compositions that can be used to treat laundry or a dishwasher load may be delivered by means of the sachet of the invention provided that the second treatment agent is water – soluble or water – dispersible. Examples include detergents, bleaches, rinse conditioners, enzymes, deodorants and rinse aids. In fabric washing, for example, a detergent composition may be delivered from the first compartment and a bleach or rinse conditioner from the second; such a product may be a self – contained whole wash product. An additive product intended to boost the performance of a conventional detergent powder may, for example, deliver an enzyme from the first compartment and a bleach from the second. A product for dishwasher use may, for example, deliver a cleaning composition with enzyme from the first compartment and a chlorine bleach from the second. These are only examples, and the skilled worker will readily be able to think of other combinations where segregation combined with delayed or retarded release of certain components is advantageous or even essential.

In the present specification the terms "wash liquor", "wash process" and "wash environment" should be understood to apply both to fabric washing machines and to dishwashing machines. The expression "start of the wash process" will also be used, and this should be understood as the time at which the water fill is substantially complete and full agitation commences; any period of static heat – up and any short bursts of agitation before water fill is complete are regarded as occurring before the start of the wash process.

In principle the sachet of the invention may have any number of compartments greater than one, but for simplicity the preferred embodiment of a two – compartment sachet will now be discussed.

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The first compartment is so designed that its contents will be released at or very shortly after the beginning of the wash cycle in the washing or dishwashing machine. Substantially complete delivery of the contents of the first compartment occurs within 3 minutes of the start of the wash process, and preferably within 1 minute. The first compartment must therefore combine the ability to contain a composition securely in the dry state with the ability to release that composition quickly when exposed to water, or water and agitation, in the wash environment.

The first compartment may be provided with one or more seals that open when the sachet is exposed to the wash environment, for example, seals sensitive to water, temperature or agitation. A water-sensitive adhesive as described in GB 1 583 082 (Unilever) may be used; or a mechanically weak heat-seal as described in EP 11 500B (Unilever). An example of a suitable water-sensitive adhesive is sodium carboxymethylcellulose.

When the contents of the first compartment are liquid, it may suitably be provided with one or more mechanically weak heat – seals, as described in EP 40 931B (Unilever), that will open as a result of agitation in the wash environment.

If the first compartment is of the opening type and its contents are in powder form, it may be made of either water – permeable or water – impermeable material. Suitable materials include wet – strength paper; woven; knitted or nonwoven fabrics; and plastics films. A material that has been found to be highly suitable is tea bag paper manufactured by Crompton Ltd., UK. The impermeable materials mentioned are also suitable for containing liquids.

It is also possible for the first compartment, if its contents are in powder form, to be of a non – opening type, in which case its walls must be made of highly water – permeable material. The pore size of the wall material must be sufficiently large to allow very rapid leaching out of the contents of the compartment, but it may be necessary to prevent leakage of the contents in the dry state, for example, by ensuring that the composition contained in the compartment is free of particles smaller that the pore size of the wall material, or by coating the outside of the compartment walls with a pore – occluding coating or layer of material that will be disrupted (dissolved or dispersed) very rapidly by the wash liquor.

The second compartment, in contrast to the first, is so designed that its contents, which are in powder form, will be released only gradually and/or after a delay of at least 5 minutes. Preferably release, even when gradual, occurs only after a delay of at least 5 minutes, preferably at least 10 minutes: the delay required will depend on the intended use of the sachet of the invention and the machine cycle that it has to match, delays of from 5 to 90 minutes typically being useful. The second compartment is of the non – opening type, and therefore it has at least one wall of porous water – permeable material. Release from the second compartment is by leaching out of its contents by the wash liquor through its porous water – permeable wall(s), and this process is delayed and/or retarded by means of one or both of two measures.

The first of these is the provision of a pore – occluding coating or layer of a material that is disrupted (dissolved or dispersed) by the wash liquor; unlike the coating mentioned above for the first compartment, this should be of a material that is not too soluble or dispersible, so that its disruption occurs over a sufficiently long period to provide some delay and/or retardation.

Thus the sachet product of the invention may comprise a first compartment, and an adjacent second compartment separated from the first by a non-opening seam or a non-water-permeable partition wall, the second compartment being provided with a pore-occluding coating or layer.

Alternatively or additionally, the sachet can be enclosed within another sachet compartment of porous water – permeable material. This other sachet compartment may simply be the first compartment; the second compartment (inner sachet) is then located, together with the first treatment agent, in the first compartment (outer sachet). In this case, the first compartment (outer sachet) should be of the non – opening type to prevent escape of the inner sachet into the wash liquor, if the inner sachet does not have a pore – occluding coating or layer.

The inner sachet may be wholly separate from the outer sachet, or it may be attached; an integral construction created by folding is also possible. According to yet another possibility, the second compart—ment (inner sachet) may be located in a separate additional (third) compartment separated from the first compartment by a non—opening seam or a partition wall which is preferably water—impermeable.

The inner sachet may conveniently be made from a water-impermeable, thermoplastic sheet material, such as polyethylene film, provided with pin-holes. This form of construction is preferred as the inner sachet is readily heat-sealable. Additionally, by varying the number and size of the pin-holes the delay in release of the second treatment agent may be varied. Rapid release may be achieved by the provision of a large number of large diameter holes whereas slower release can be obtained from sachets having a small number of smaller diameter holes. Sachets for use in a conventional sized machine are preferably provided with a total of 2 to 8 pin-holes having a diameter of 0.5 to 1mm. Reproducibility of release profiles is generally increased by provision of a large number of small holes and also by the provision of pin-holes in both faces of the sachet. Reproducibility is also increased in general when the pin-holes are comparatively close to the sachet corners.

In embodiments where the first and second compartments have a porous wall in common, there is a danger of premature mixing in the dry state when the sachets are transported, stored and handled. The use of a pore – occluding coating or layer for the second compartment is then particularly beneficial. When no such coating or layer is to be used, designs in which the two compartments are separated by a water – impermeable partition or by a non – opening seam are preferred.

If the second compartment is integral with the first it is most conveniently made of the same material, which will of course have the same porosity. This is, however, not essential. If the first compartment is of the opening type, the porosity of that material can be relatively low, and a coating to reduce the porosity of the second compartment may be unnecessary. If the first compartment is non-opening and of relatively high porosity, a coating will probably be needed for the second compartment and a different (more quickly disrupted) coating may also be needed for the first compartment.

If the second compartment is a separate inner sachet, it may of course be made of a different material from that of the first compartment and the porosity of both compartments can then be chosen at will.

Examples of suitable pore – occluding coating materials include fatty acids, for example, stearic acid, which disperse slowly; polyethylene glycols, which can disperse quickly or slowly depending on molecular weight; mixtures of fatty acids and polyethylene glycol, which can be tailored by suitable choice of proportions to give any chosen release rate; and long – chain nonionic surfactants, for example, tallow alcohol ethoxylates. A separate discrete layer of pore – occluding material, for example, polyvinyl alcohol film, may instead be laminated onto the porous wall material.

The pore – occluding material is preferably coated onto the sachet material at a level of 50 to 300g/m², more preferably 150 to 250g/m². The release rate is conveniently tailored by using a mixture of materials. For example, a 80:20 mixture of tallow 18EO and stearic acid will give a much greater release rate than a 20:80 mixture of the same components. In general, the rate of release is enchanced by the use of a high propertion of a material which is dispersed quickly in the wash liquor and depressed by the use of a material which is dispersed slowly. The skilled worker will be able to determine suitable proportions by simple experimentation.

Description of preferred embodiments

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Embodiments of the invention will now be described, by way of example only, with reference to the accompanying drawings in which:

Figure 1 is a plan view of a first sachet in accordance with the invention;

Figure 2 is a section, on a larger scale, along the line II - II of Figure 1;

Figure 3 is a plan view of a second sachet in accordance with the invention;

Figure 4 is a section, on a larger scale, along the line IV - IV of Figure 3;

Figure 5 is a plan view of a third sachet in accordance with the invention;

Figure 6 is a section, on a larger scale, along the line VI - VI of Figure 5;

Figure 7 is a plan view of a fourth sachet in accordance with the invention; and

Figure 8 is a schematic sectional view, on a larger scale, of a sachet as shown in Figure 7.

Referring now to Figure 1 of the accompanying drawings, a two-compartment sachet 1 has a large first compartment 2 and a smaller second compartment 3. The sachet is made, for example, of Crompton (Trade Mark) tea bag paper, a cellulosic material which contains thermoplastic (polypropylene) fibres to render it heat-sealable. The second compartment 3 is bounded by strong heat-seals 4 that will not open in the wash liquor, while the remaining three edge seals 5 of the first compartment 2 are formed by a water-soluble adhesive, for example, sodium carboxymethylcellulose.

Referring now to Figure 2, the second compartment 3 is coated externally with a coating or layer 6 of pore – occluding material, for example, tallow alcohol 18 EO. The first compartment 2 contains a first powdered composition 7, for example, a detergent powder with enzyme, and the second compartment 3 contains a second powdered composition 8, for example, a bleach. In use, the sachet is placed together with the load in a washing or dishwashing machine. The first compartment opens very rapidly along the edges 5 and releases its contents within 1 to 3 minutes. The coating 6 on the second compartment is gradually dissolved and after a delay of about 5 – 15 minutes the walls of the compartment 3 are sufficiently exposed for its contents 8 to be leached out by the wash liquor.

Figure 3 and 4 of the accompanying drawings show a sachet of slightly different construction. The second compartment 3 takes the form of an inner sachet within a third compartment 9. The inner sachet 3 may be of the same or a different material to that of the main sachet 1.

A different sachet construction is shown in Figures 5 and 6 of the accompanying drawings. This sachet 10 is produced from a single sheet of porous water – impermeable material, for example, the polypropylene nonwoven fabric Kimtex (Trade Mark) ex Kimberly – Clark. The sheet is folded so as to generate a small inner compartment 11 (the second compartment) within a principal compartment 12 (the first compart – ment), and closed by heat – sealing along the folded edge 13 and along the other three edges 14: the edge seals 14 may be opening or non – opening. If desired the external surfaces of the walls of the inner compartment 11 could be coated or laminated with a pore – occluding material, but it is possible to obtain suitable delivery characteristics without using such a coating or layer if a sheet material of appropriate porosity is chosen. Alternatively, by using a sheet made of a mixture of materials joined for example by glueing or welding, the first compartment may be made of material different from the second.

As in the other embodiments the base weight of the sheet material is not critical, preferably it is in the range 15 to 150g/m². If the material has a very high base weight some difficulty may be experienced in heat sealing the sachet as in some parts the construction is four layers thick, but the problem may be overcome by glueing.

Yet another sachet design is shown in Figures 7 and 8 of the accompanying drawings. An outer sachet 15 (the first compartment) of porous water – permeable sheet material contains the first powdered com – position 7 for rapid delivery to the wash liquor and also contains an inner sachet 16 (the second compartment), also of water – permeable sheet material, containing the second powdered composition 8. As shown the inner sachet 16 is loose within the outer sachet 15, but if desired its position could be fixed, for example, by means of an edge seal common to both sachets. If desired, the inner sachet 16 may be provided with a pore – occluding outer coating or layer, but as with the embodiment of Figures 5 and 6 this may not be necessary if the porosities of the materials of the sachets (which may of course be different) are suitably chosen.

In any of the embodiments described above, the sachet product of the invention may be of such a size than a single unit will deliver an appropriate quantity of ingredients for a single washload. Greater flexibility for the consumer is, however, achieved if smaller units are produced in groups, for example, of two to six units, readily separable by tearing along perforated marginal regions.

EXAMPLE 1

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An experiment was carried out to determine the release times of the two compartments of the sachet described above with reference to Figures 3 and 4 of the accompanying drawings. The main sachet 1 was made of Crompton (Trade Mark) 784 tea bag paper ex Crompton, and had overall dimensions of 15 cm x

15 cm. The first compartment contained 110.6 g of non-bleaching detergent powder, including 3.6 g of sodium bromide, and was sealed with detergent-grade sodium carboxymethylcellulose. The inner sachet 3, of Kimtex (Trade Mark) polypropylene nonwoven fabric ex Kimberly-Clark, had dimensions of 13 cm x 5cm and contained 18 g of potassium peroxomonosulphate triple salt ex Interox. The inner sachet 3 had an outer coating of 200 g/m² (2.6 g in total) of tallow alcohol 18 EO (Lutensol (Trade Mark) AP 18 ex BASF).

Nine washing machine experiments were carried out to determine the time taken for the inner sachet 3 to release it contents. A Miele (Trade Mark) De Luxe Electronic 756 washing machine set to the 40°C economy programme was used, with a mixed load of 2.5 kg of clean cotton and synthetic fabrics.

In each run the detergent powder was completely delivered from the first compartment within a period of 30 seconds to 1 minute from the start of the wash process. Delivery of substantially all of the bleaching agent from the second compartment took place in each run after a delay of 5 to 10 minutes, as can be seen from the Table below. This period can be lengthened or shortened by varying the type and level of coating on the inner sachet 9.

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	Bleach released					Time (Mins)		
	(gra	ams)	1	3	5	77	10	15
20	Run	1	0	0	1.4	15.6	18.0	_
	Run	2	0	0.2	2.1	18.0	18.0	
	Run	3	0	0	4.0	12.8	18.0	_
25	Run	4	0	0	3.6	18.0	18.0	-
	Run	5	0	0.4	1.7	18.0	18.0	-
	Run	6	0	0	0.9	15.4	18.0	•
30	Run	7	0	0	0	2.4	15.6	18.0
	Run	8	0	0	0	3.4	17.1	18.0
	Run	9	0	0	0	2.1	13.0	18.0

EXAMPLE 2

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A sachet of the type described by reference to Figures 7 and 8 was prepared as follows.

A rectangle of polyethylene film (6cm by 3cm) of 115µm thickness was folded along the minor axis and heat – sealed along two edges. Calcium hypochlorite (1.25g) was placed in the sachet which was then sealed along the open edge to give a square sachet having 3cm sides. Three 1mm holes were made in each face of the sachet.

A square sachet having 10cm sides was made from a sheet of Sontara (Trade Mark) 8000 non – woven polyester sheet by folding a sheet of 10 x 20cm material. Two sides were heat – sealed, and 25g of conventional washing powder and the 3 x 3cm sachet placed inside. The final seam was closed by heat – sealing. The complete sachet was placed in a Miele (Trade Mark) 429 washing machine along with a 2.5kg ballast load of terry towelling and cotton sheeting. The machine was set into action to run a 30 °C cycle and the concentration of hypochlorite in the wash liquor measure at regular intervals. The following results were obtained:

	Time	(mins)	Conc ⁿ of hypochlorite in wash liquor	(ppm)
5	5		0	
	10		0	
	15		23.9	
10	20		81.6	
, -	25		97.6	
	Rinse	1	29.3	
		2		
15		_	14.2	
		3	12.3	
		4	0.9	
		5	0	
20				

The detergent was released within 3 minutes but release of the bleach was delayed for almost 15 minutes.

25 Example 3

A sachet of the type described by reference to Figure 1 and 2 was prepared as follows: A strip of polyethylene laminated cellulosic non-woven fabric of base weight $30g/m^2$) (Storalene (Trade Mark)) of dimensions 30×5 cm was folded along the minor axis so that the polyethylene coated sides were adjacent to each other. The long seams were then heat – sealed. 25g of conventional washing powder was introduced into the sachet which was then heat – sealed so that the powder was confined in a compartment of dimensions 15×5 cm defined in part by the folded seam. Calcium hypochlorite (1.25g) was introduced into the open end of the sachet and the sachet sealed. Two pin – holes (1mm diameter) were made in each face of the hypochlorite containing compartment, which compartment was then coated at $200g/m^2$ with a polyethylene glycol having an average molecular mass of 35,000.

The rate of release of the hypochlorite was measured in a similar experiment to that described in Example 2. The following results were obtained:

40	Time mins)	Conc ⁿ of hypochlorite in wash liquor (ppm)
	5	0
45	10	0
	15	0
	20	17.7
50	25	24.9
50	30	25.0

In this case no detectable amount of bleach was released for more than 15 minutes and the detergent composition was released within 3 minutes.

Example 4

A piece of Kimtex (Trade Mark) melt blown polypropylene non – woven fabric of 70g/m² and dimensions 15 x 27cm was folded along the minor axis. Two further folds, equidistant (7.5cm) from the first and parallel with it were then made so that the fabric adopted an 'M' shaped configuration. Three of the seams were then heat – sealed to give two open compartments, one of which was contained within the other. The inner compartment was filled with potassium monopersulphate (3g) and the outer compartment was filled with a conventional detergent (20g) and sodium bromide (0.6g). The remaining seam was heat sealed with the rate of release of bleach into wash liquor determined as described in Examples 2 and 3. The following results were obtained:

	Time (mins)	Amount of bleach released (g)
15		
	0.5	0
	6.0	1
20	10	1.6
	15	3.0
	20	3.0

The delay of release was less than in the previous examples; about half the bleach was released with 10 minutes and all within 15 minutes. The detergent was released immediately.

Claims

- 1. A product for the delivery of treatment agents into the wash liquor of an automatic washing or dishwashing machine, comprising a sachet having at least two compartments, including:
 - (i) a first compartment of water-insoluble material containing a first treatment agent, said first compartment having at least one opening seal and/or being formed of porous water-permeable material, and being capable of releasing said first treatment agent into the wash liquor of a washing or dishwashing machine within a period of 3 minutes from the start of a wash process carried out with wash water at 30°C; and
 - (ii) a second, non opening, compartment containing a second, water soluble or water dispersible, treatment agent in particulate form, said second compartment being formed at least partially of porous water permeable material through which said second treatment agent can be leached out by the wash liquor, said second compartment being provided with means for retarding release from the second compartment so as to take place more gradually than release from the first compart ment, and/or delaying release from the second compartment for at least 5 minutes from the start of a wash process carried out with wash water at 40 °C, said means for retarding or delaying release comprising
 - (a) a substantially wholly pore-occluding external coating or layer that is capable of being disrupted by the wash liquor, and/or
 - (b) the enclosure of the second compartment within another sachet compartment of porous water permeable material.
- 2. A product as claimed in claim 1, wherein the second compartment is adjacent to the first compartment and separated therefrom by a non-opening seam, the walls of the second compartment being provided with a pore-occluding coating or layer.
- 3. A product as claimed in claim 1, wherein the second compartment is a separate sachet located within the first compartment.

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- 4. A product as claimed in claim 1, wherein the second compartment is integral with the first and located therein by means of folding.
- 5. A product as claimed in claim 1, wherein the second compartment is a separate sachet located within a third compartment separated from the first compartment by a non opening seam.
- 6. A product as claimed in any one of claims 3 to 5, wherein the walls of the second compartment are provided with a pore occluding coating or layer.

10 Patentansprüche

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- 1. Produkt für die Abgabe von Behandlungsmitteln in die Waschflüssigkeit einer automatischen Wasch oder Geschirrspülmaschine, umfassend einen Beutel mit wenigstens zwei Kammern, einschließend:
 - i) eine erste Kammer aus wasserunlöslichem Material, enthaltend ein erstes Behandlungsmittel, wobei die erste Kammer wenigstens eine sich öffnende Verklebung besitzt und/oder aus einem porösen, wasserdurchlässigen Material gebildet ist, und in der Lage ist, das erste Behandlungsmittel in die Waschflüssigkeit einer Wasch oder Geschirrspülmaschine innerhalb eines Zeitraums von drei Minuten nach dem Beginn eines Waschprozesses, durchgeführt mit warmem Wasser bei 30°C, freizusetzen; und
 - ii) eine zweite, sich nicht öffnende Kammer, enthaltend ein zweites, wasserlösliches oder wasser dispergierbares Behandlungsmittel in Form einzelner Teilchen, wobei die zweite Kammer wenigstens teilweise aus wasserdurchlässigem Material gebildet ist, durch das das zweite Behandlungsmittel durch die Waschflüssigkeit ausgelaugt werden kann, wobei die zweite Kammer mit Mitteln zur Verlangsamung der Freisetzung aus der zweiten Kammer versehen ist, um diese länger als die Freisetzung aus der ersten Kammer allmählich durchzuführen, und/oder zur Verzögerung der Freisetzung aus der zweiten Kammer für wenigstens fünf Minuten nach dem Beginn des Wasch prozesses, durchgeführt mit Waschwasser bei 40°C, wobei die Mittel zur Verlangsamung oder Verzögerung der Freisetzung umfassen
 - (a) eine im wesentlichen vollständig Poren abschließende, äußere Beschichtung oder Schicht, die in der Lage ist, durch die Waschflüssigkeit zerstört zu werden, und/oder
 - (b) den Einschluß der zweiten Kammer innerhalb einer weiteren Beutelkammer aus porösem wasserdurchlässigem Material.
- 2. Produkt nach Anspruch 1, worin die zweite Kammer der ersten angrenzend ist und durch diese durch eine nicht öffnende Naht getrennt ist, wobei die Wände der zweiten Kammer mit einer Poren abschließenden Beschichtung oder Schicht versehen sind.
 - 3. Produkt nach Anspruch 1, worin die zweite Kammer ein innerhalb der ersten Kammer befindlicher separater Beutel ist.
 - 4. Produkt nach Anspruch 1, worin die zweite Kammer mit der ersten intregiert ist und darin mittels Falten plaziert ist.
- 5. Produkt nach Anspruch 1, worin die zweite Kammer ein getrennter Beutel ist, der sich innerhalb einer dritten Kammer befindet, die von der ersten Kammer über eine nicht öffnende Naht getrennt ist.
 - 6. Produkt nach einem der Ansprüche 3 bis 5, worin die Wände der zweiten Kammer mit einer Poren abschließenden Beschichtung oder Schicht versehen sind.

50 Revendications

- 1. Un produit pour la distribution d'agents de traitement dans la liqueur de lavage d'une machine à laver ou d'une machine à laver la vaisselle automatique, comprenant un sachet ayant au moins deux compartiments, incluant :
 - (i) un premier compartiment en matériau insoluble dans l'eau contenant un premier agent de traitement, ledit premier compartiment ayant au moins une fermeture ouvrante et/ou étant formé d'un matériau poreux perméable à l'eau, et étant capable de libérer ledit premier agent de traitement dans la liqueur de lavage d'une machine à laver ou d'une machine à laver la vaisselle en une

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période de 3 minutes à partir du début d'un traitement de lavage effectué avec une eau de lavage à 30°C; et

- (ii) un second compartiment ne s'ouvrant pas contenant un second agent de traitement, soluble dans l'eau ou dispersible dans l'eau, sous forme particulaire, ledit second compartiment étant formé au moins partiellement de matériau poreux perméable à l'eau hors duquel lequel ledit second agent de traitement peut être lessivé par la liqueur de lavage, ledit second compartiment étant équipé de moyens pour retarder la libération hors du second compartiment de manière à ce qu'elle ait lieu de manière plus graduelle que la libération hors du premier compartiment, et/ou pour différer la libération hors du second compartiment pendant au moins 5 minutes à partir du début d'un traitement de lavage effectué avec une eau de lavage à 40°C, lesdits moyens pour retarder ou différer la libération comprenant
 - (a) une couche ou un enrobage externe obstruant de manière pratiquement' complète les pores qui est susceptible d'être désintégrée par la liqueur de lavage, et/ou
 - (b) l'inclusion du second compartiment à l'intérieur d'un autre compartiment du sachet en matériau poreux perméable à l'eau.
- 2. Un produit selon la revendication 1, dans lequel le second compartiment est adjacent au premier compartiment et séparé de celui-ci par une jonction ne s ouvrant pas, les parois du second compartiment étant dotées d'une couche ou d'un enrobage d'occlusion des pores.
- 3. Un produit selon la revendication 1, dans lequel le second compartiment est un sachet séparé situé à l'intérieur du premier compartiment.
- 4. Un produit selon la revendication 1, dans lequel le second compartiment fait partie intégrante du premier et se trouve situé dedans au moyen d'un pliage.
 - 5. Un produit selon la revendication 1, dans lequel le second compartiment est un sachet séparé situé à l'intérieur d'un troisième compartiment séparé du premier compartiment par une jonction ne s'ouvrant pas.
- Un produit selon l'une quelconque des revendications 3 à 5, dans lequel les parois du second compartiment sont dotées d'une couche ou d'un enrobage d'occlusion des pores.

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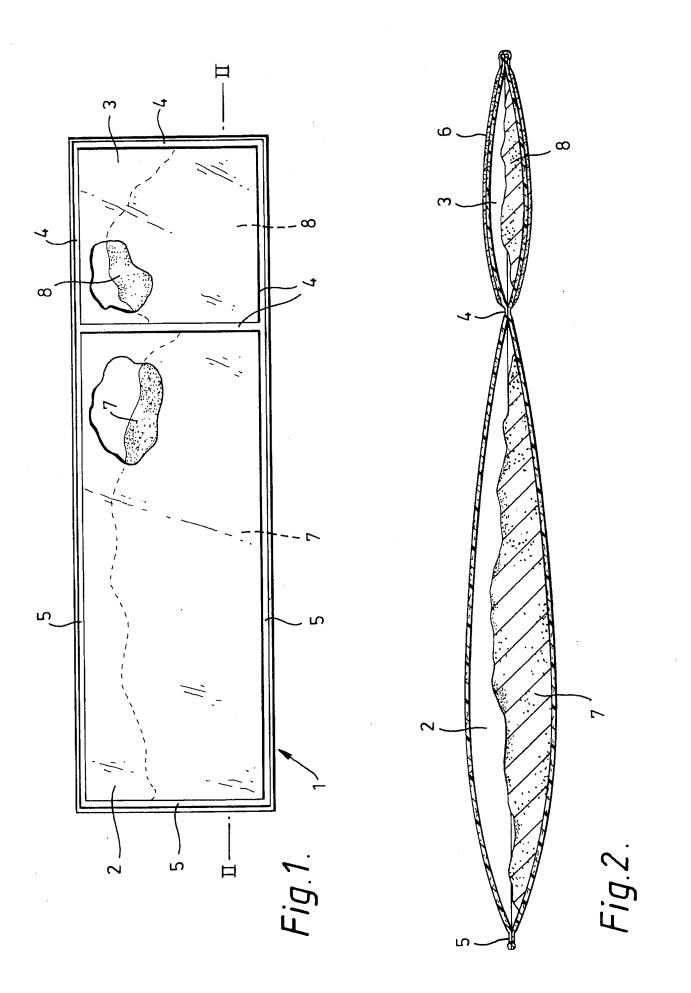
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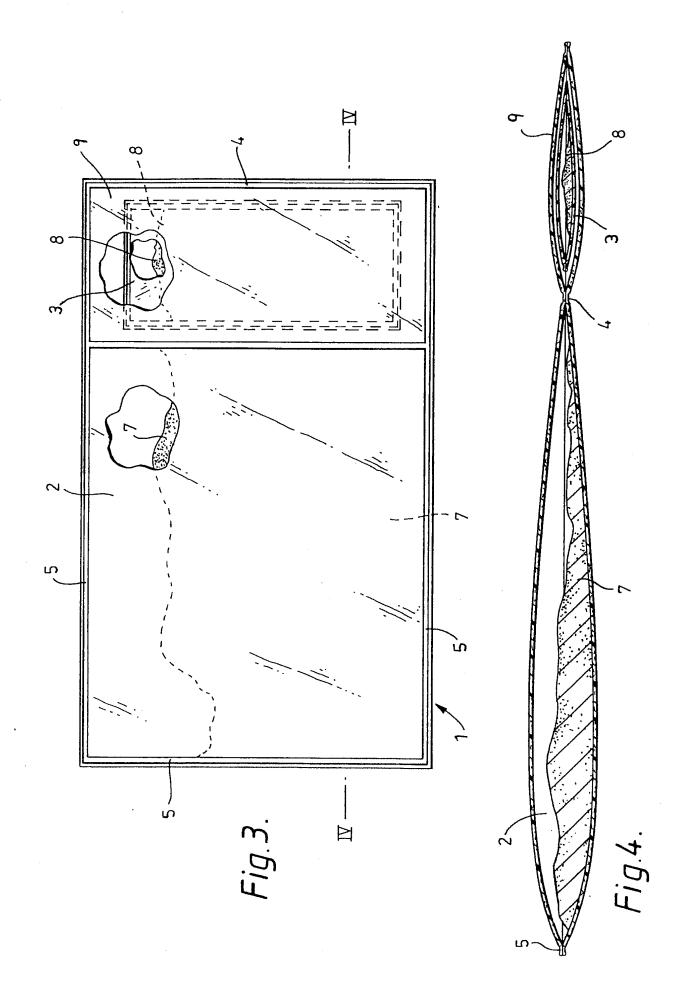
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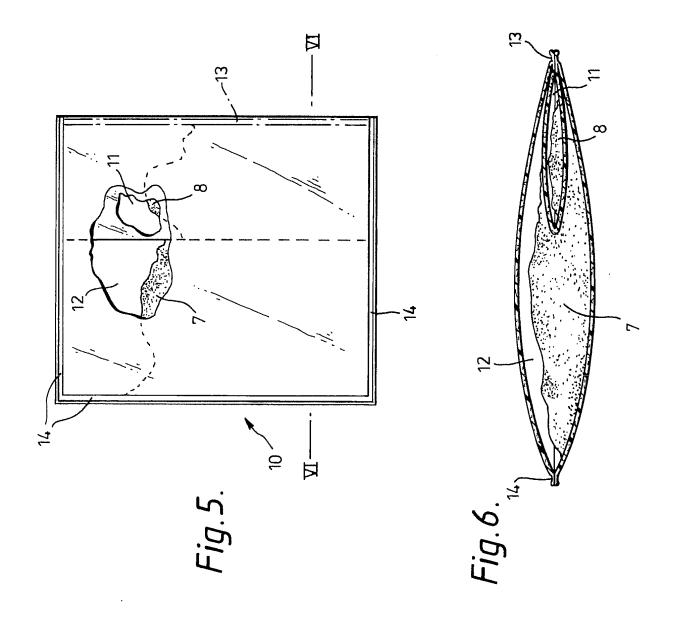
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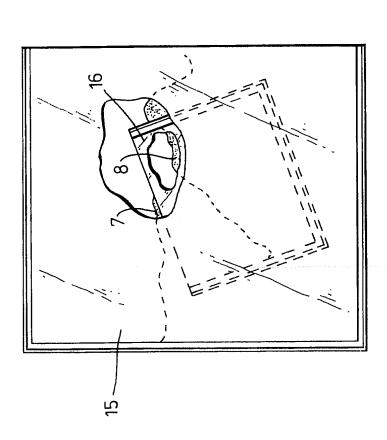
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Fig. 7.

(11) Veröffentlichungsnummer:

0 132 792 A1

(12)

EUROPÄISCHE PATENTANMELDUNG

21 Anmeldenummer: 84108518.6

(51) Int. Cl.4: C 11 D 3/37

22) Anmeldetag: 19.07.84

30 Priorităt: 20.07.83 DE 3326091

(43) Veröffentlichungstag der Anmeldung: 13.02.85 Patentblatt 85/7

Benannte Vertragsstaaten: AT BE CH DE FR GB IT LI NL Anmeider: DISPO-Kommerz AG Sonnhaldenstrasse 15 CH-6331 Hünenberg/Zug(CH)

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Wasserlösliche, pulverförmige Reinigungsmittel für harte Oberflächen.

Tur Reduzierung des Wasser- und Heizenergieverbrauchs insbesondere bei Spülmaschinen wird ein wasserlösliches, pulverförmiges Reingungsmittel vorgeschlagen, welches auch bei erhöhtem Fließwiderstand und stärkerem Schmutzanfall eine gute Reinigungswirkung aufweist. Dieses wird durch ein Reinigungsmittel für harte Oberflächen, insbesondere von Geschirr, Eßbestecken und anderen Küchengeräten, in automatischen Spülmaschinen, auf Basis von alkalischen Trägermaterialien, alkalibeständigen nichtionischen Tensiden und üblichen Zusätzen, wie Bleichmitteln (Peroxidverbindungen), Biociden, Duftstoffen, Schaumverhütungsmitteln und/oder Lösungsvermittlern erreicht, das gekennzeichnet ist durch einen Gehalt an Polycarbonsäuren und/oder deren Salzen mit einem Molekulargewicht von 12 000 bis 40 000.

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Wasserlösliche, pulverförmige Reinigungsmittel für harte Oberflächen.

Die Erfindung betrifft wasserlösliche, pulverförmige Reinigungsmittel für harte Oberflächen, die insbesondere für Spülmaschinen geeignet sind.

Für wasserlösliche, pulverförmige Reinigungsmittel ist aus der US-PS 3 922 230 die Mitverwendung von Alkyli- . metall-, Ammonium- oder substituierten Ammoniumsalzen von Polyacryl- oder Polymethacrylsäuren mit einem Molekulargewicht von 500 bis 10 000, vorzugsweise von unter 10 5000, als Gerüststoffe bzw. Waschmittelverstärker, sogenannte Builder, bekannt, die allein oder in Verbindung mit anderen Buildersubstanzen in den Reinigungsmitteln verwendbar sind. Von dem Einsatz höhermolekularer Polyacryl- oder Polyalkylacrylsäuresalzen, wie sie beispiels-15 weise in der GB-PS 1 090 809 oder in der US-PS 2 327 302 genannt sind, wird abgeraten, da derartige höhermolekulare und/oder halogensubstituierte Polymerisate biologisch nicht abbaubar seien.

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Ein weiteres Problem, das insbesondere bei den mit den bekannten Reinigungsmitteln betriebenen Spülmaschinen auftritt, ist der relativ hohe Verbrauch an Wasser und Heizenergie. Um hier Einsparungen erzielen zu können, ist es bekannt, den Kreislauf der Reinigungsmittellösung durch Verwendung von Mikrosieben zu verengen. Doch wird hierdurch der Fließwiderstand der Lösung erhöht und die Reinigungskraft der Reinigungsmittellösung verringert.

- Der Erfindung liegt die Aufgabe zugrunde, ein Reinigungsmittel zu schaffen, das auch bei erhöhter Schmutzlast,
 die beispielsweise durch eine Verengung der Zufluß- und
 Abflußkanäle oder durch Reduzierung der Spülflotte verursacht wird, eine einwandfreie Reinigung von harten
 Oberflächen, insbesondere von Geschirr, Eßbestecken und
 anderen Küchengeräten, ermöglicht. An derartige Reinigungsmittel werden folgende Anforderungen gestellt:
- a) Sie sollen im stark alkalischen Medium, d.h. bei
 pH-Werten von 11 bis 12, stabil sein;
 - b) Sie sollen ökologisch unbedenklich und ungiftig sein;
 - c) Sie sollen mit den anderen chemischen Substanzen des Reinigungsmittels verträglich sein.
- Diese Aufgabe wird gelöst von wasserlöslichen, pulverförmigen Reinigungsmitteln für harte Oberflächen, insbesondere von Geschirr, Eßbestecken und anderen Küchengeräten, in automatischen Spülmaschinen, auf Basis von
 alkalischen Trägermaterialien, alkalibeständigen nichtionischen Tensiden und üblichen Zusätzen, wie Bleichmitteln (Peroxidverbindungen), Biociden, Duftstoffen,
 Schaumverhütungsmitteln und/oder Lösungsvermittlern, die

gekennzeichnet sind durch einen Gehalt an Polycarbonsäuren und/oder deren Salzen mit einem Molekulargewicht von 12 000 bis 40 000, vorzugsweise von 15 000 bis 28 000 und bevorzugt von 19 000 bis 22 000.

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Die Polycarbonsäuren und/oder ihre Salze sind in einer Menge von 1 bis 8, vorzugsweise 2,5 bis 4 Gewichtsprozent, bezogen auf das Gesamtgewicht des Reinigungsmittels, vorhanden.

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Die Polycarbonsäuren weisen zweckmäßigerweise etwa 100 Carboxylgruppen im Molekül auf, insbesondere wenn sie in Form ihrer Salze eingesetzt werden. Bevorzugt werden solche Polycarbonsäuren, bei denen zwischen zwei Carboxylgruppen 1 bis 3, vorzugsweise2 Kohlenstoffatome stehen.

Als Polycarbonsäuren kommen in erster Linie Polyacrylsäuren, Polymethacrylsäuren und Polymerisationsprodukte
von Maleinsäure und bzw. oder deren Anhydrid und/oder
Fumarsäure und Athylen und/oder Propylen in Betracht.
Bevorzugte Salze dieser Polycarbonsäuren sind die Alkalimetall-, Ammonium- und substituierten Ammoniumsalze, insbesondere die Natriumsalze. Als Substituenten bei den
substituierten Ammoniumsalzen werden Alkylgruppen mit
l bis 3 Kohlenstoffatomen, die als weiteren Substituenten
Hydroxylgruppen aufweisen können, bevorzugt.

Die in den Reinigungsmitteln nach der Erfindung enthaltenen alkalischen Trägermaterialien sind als solche bekannt und schließen wasserlösliche Citrate, wie Trinatriumcitrat, und Silikate, wie Natriummetasilicate, sowie Alkalicarbo-

nate und -hydroxide ein.

Als alkalibeständige nicht-ionische Tenside haben sich als vorteilhaft Athylen- und/oder Propylenoxidanlagerungsprodukte an Fettalkohole mit 12 bis 18 Kohlenstoffatomen oder an Oxoalkohole mit 10 bis 15 Kohlenstoffatomen erwiesen. Besonders bevorzugt sind Anlagerungsprodukte von 6 bis 8 Mol Athylenoxid und 3 bis 6 Mol Propylenoxid an Alkohole mit 12 bis 15 Kohlenstoffatomen.

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Den Reinigungsmitteln können je nach Belieben übliche Zusatzstoffe wie Bleichmittel, insbesondere Peroxidverbindungen, wie Percarbonate, Perborate oder Magnesiumperoxyphthalat, ferner Duftstoffe, übliche Biocide, Schaumverhütungsmittel, Puffersubstanzen und/oder Lösungsvermittler wie Natriumsulfat, in üblichen Mengen zugegeben werden.

Die Verwendung der Polycarbonsäuren und/oder deren Salze verbessert die Schmutzlösung, das Schmutzdispergierver20 mögen und das Fließvermögen derartig, daß unter normalen Spülflottenverhältnissen eine stark verbesserte Reinigungskraft und sogar bei einer reduzierten Spülflotte und bei einem verkleinerten Laugenkreislauf die normale Reinigungskraft erhalten oder sogar verbessert wird. Ein weiterer
25 Vorteil ist darin zu sehen, daß der Zusatz einer Polycarbonsäure und/oderderen Salzen mit den genannten Molekulargewichten die Anwendung niedrigerer Spültemperaturen gestattet, als dies bisher der Fall war.

Die geschilderten Vorteile werden auch dann erhalten, wenn man den Reinigungsmitteln gegebenenfalls weitere Buildersubstanzen, wie die üblichen Tripolyphosphate (Pentanatriumoder Pentakalium-tripolyphosphate) oder Alkalipyrophosphate, zugibt. Wenn es die ökologischen Bedingungen gestatten, können auch chlorabspaltende Substanzen, wie das Natriumsalz der Dichlor-isocyanursäure oder 1,3-Dichlor-5,5-dimethylhydantoin (das unter dem Warenzeichen "Halane" von der Firma Wyandotte Chemicals Corporation, V.St.A., vertrieben wird) in den Reinigungsmitteln der Erfindung mitverwendet werden.

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Einen weiteren Gegenstand vorliegender Erfindung bildet ein Verfahren zur Herstellung der Reinigungsmittel, das dadurch gekennzeichnet ist, daß man in einer Mischvorrichtung, vorzugsweise einem Pulvermischer, ein alkalibeständiges nichtionisches Tensid auf ein alkalisches Trägermaterial aufsprüht und unmittelbar nach dem Aufsprühen des Tensids eine Polycarbonsäure und/oder deren Salz mit einem Molekulargewicht von 12 000 bis 40 000 zugibt.

Vorzugsweise wird das Tensid in die Mischvorrichtung über 20 eine Winkeldüse von 60° bis 140°, vorzugsweise 70° bis 95°, senkrecht zur Mischrichtung aufgesprüht.

Auf diese Weise wird die an sich stark hygroskopische Polycarbonsäure und/oder deren Salz mittels des Tensids in das Trägermaterial eingezogen und dort versiegelt, so daß man ein frei fließendes Pulver erhält, das keine Verklumpungen oder Krustenbildungen beim Abfüllen verursacht. Außerdem wird ein Entmischen beim Lagern der Reinigungsmittel vermieden.

Nach dem Einbringen der Polycarbonsäure und/oder deren Salz können die übrigen Substanzen der Mischvorrichtung zugeführt werden. Weitere vorteilhafte Verfahren werden in den Beispielen 8, 9 und 10 angegeben. Nachstehend sind Grundrezepturen für die Reinigungsmittel nach der Erfindung aufgeführt:

5	1)	Natrium-citrat	10	-	30	GewProzent
		Natriummetasilikat (wasserfrei und/ oder hydratwasserhaltig)		-	60	GewProzent
		Polycarbonsäure vom Molekular- gewicht etwa 20 000	1		8	GewProzent
10		Natriumpercarbonat	5	-	30	GewProzent
		Tensid	1	-	3	GewProzent

Anstelle von Natriumpercarbonat kann man auch 5 bis 25 Gewichtsprozent Natriumperborat oder 3 bis 10 Gewichtsprozent Magnesiummonoperoxyphthalat verwenden.

2) Natriummetasilicat (wasserfrei und/
oder hydratwasserhaltig) 40 - 70 Gew.-Prozent
Pentanatriumtripolyphosphat 15 - 25 Gew.-Prozent
Tensid 1 - 3 Gew.-Prozent
Polyacrylsäure vom ungefähren
Molekulargewicht 20 000 1 - 8 Gew.-Prozent

Als weitere Zusätze können 0,5 bis 1,5 Gewichtsprozent chlorabspaltende Substanzen und/oder 5 bis 10 Gewichtsprozent Peroxidverbindungen zugegeben werden. Das Tripolyphosphat kann bis zu 10 Gewichtsprozent durch Natriumcitrat ersetzt werden.

Die Beispiele erläutern die Erfindung.

Beispiel 1

Reiniqungsmittel für Geschirrspülmaschinen:

5 Trinatriumcitrat 25 Gew.-Prozent

Polyacrylsäure vom mittleren

Molekulargewicht 20 000 3 Gew.-Prozent

Natriumpercarbonat 10 Gew.-Prozent

Anlagerungsprodukt von 6 bis 8 Mol

10 Athylenoxid und 3 bis 6 Mol Propylenoxid an Fettalkohole mit 12 bis 15

Kohlenstoffatomen 1 Gew.-Prozent

Natriummetasilicat (wasserfrei und

hydratwasserhaltig) ad 100 Gew.-Prozent

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Beispiel 2

Die Rezeptur des Beispiels 1 wird wiederholt, jedoch anstelle von Natriumpercarbonat werden 8 Gewichtsteile Natriumperborat eingesetzt.

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Beispiel 3

Die Rezeptur des Beispiels 1 wird wiederholt, jedoch anstelle von Natriumpercarbonat werden 5 Gewichtsprozent Magnesiummonoperoxyphthalat eingesetzt.

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Beispiel 4

Pentanatriumtripolyphosphat 22,5 Gew.-Prozent Polyacrylsäure vom Molekular-gewichtsbereich 16000-25000 3 Gew.-Prozent

30 Natriumsalz der Dichlorisocyanursäure

1 Gew.-Prozent

Anlagerungsprodukt von etwa 8 Mol Athylenoxid und 5 Mol Propylenoxid an Oxalalkohole mit 11 bis 15 Kohlenstoffatomen

1 Gew.-Prozent

Natriummetasilicat (wasserfreiund hydratwasserhaltig)

ad 100 Gew.-Prozent

Beispiel 5

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Das Beispiel 4 wird wiederholt, jedoch enthält die Rezeptur anstelle von Pentanatriumtripolyphosphat ein Gemisch aus gleichen Teilen Pentanatriumtripolyphosphat und Trinatriumcitrat sowie zusätzlich 6 Gewichtsprozent Natriumpercarbonat

Beispiel 6

Die Rezeptur des Beispiels 5 wird wiederholt, jedoch mit 15 einem Gehalt von 0,5 Gewichtsprozent 1,3-Dichlor-5,5dimethylhydantoin anstelle von 1 Gewichtsprozent des Natriumsalzes der Dichlor-isocyanursäure.

Beispiel 7

Ein Pulvermischer wird mit 500 Gewichtsteilen Trägermaterial (Natriumcitrat und/oder Natriummetasilicate) beschickt und in Rotation versetzt. Aus einer Winkeldüse, die unter einem Winkel von 80° senkrecht zur Mischrich tung angeordnet ist, wird die erforderliche Menge Tensid auf das Trägermaterial aufgesprüht. Unmittelbar nach dem Aufsprühen und bevor das Tensid in das Trägermaterial eingedrungen ist, wird die Polycarbonsäure in der gewünschten Menge in den Pulvermischer gegeben. Nachdem die Polycarbonsäure mit Hilfe des Tensids in das Trägermaterial eingezogen ist, werden die übrigen Bestandteile der gewünschten Rezeptur in den Pulvermischer eingebracht.



Es wird solange gemischt, bis ein homogenes Pulver entstanden ist, das dann dem Pulvermischer entnommen und abgepackt wird.

Diese Arbeitsweise hat den Vorteil, daß die start hygroskopische Polycarbonsäure in dem Trägermaterial mittels des Tensids versiegelt vorliegt und dadurch vor dem Angriff von Luftfeuchtigkeit geschützt ist.

10 <u>Beispiel 8</u>

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In einem separaten Pulvermischer werden 60 Gewichtsteile Polycarbonsäure und 20 Gewichtsteile Tensid vorgemischt, bis die Mischung eine trockene Konsistenz aufweist. Danach wird dieser Premix in den Hauptmischer zu ca. 500 Gewichtsteilen Trägermaterial (z.B. Pentanatriumtripolyphos phat, Trinatriumcitrat und/oder Natriumsilikat) gegeben.

Beispiel 9

In einen separaten Pulvermischer gibt man 50 Gewichtsteile Trägermaterial, 60 Gewichtsteile Polycarbonsäure und
Gewichtsteile Tensid und mischt diese Substanzen bis zur
trockenen Konsistenz.

Dieser Premix wird in den Hauptmischer zu 450 Gewichtsteilen des alkalischen Trägermaterials (z.B. Pentanatrium-tripolyphosphat, Trinatriumcitrat und/oder Alkalisilicat) gegeben und gut eingemischt. Anschließend werden die restlichen Bestandteile zugemischt.

Patentansprüche

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Wasserlösliche, pulverförmige Reinigungsmittel für harte Oberflächen, insbesondere von Geschirr, Eβbestecken und anderen Küchengeräten, in automatischen Spülmaschinen, auf Basis von alkalischen Trägermaterialien, alkalibeständigen nicht-ionischen Tensiden und üblichen Zusätzen, wie Bleichmitteln (Peroxidverbindungen), Biociden, Duftstoffen, Schaumverhütungsmitteln und/oder Lösungsvermittlern,

gekennzeichnet durch

einen Gehalt an Polycarbonsäuren und/oder deren
Salzen mit einem Molekulargewicht von 12 000 bis
40 000.

- Reinigungsmittel nach Anspruch 1, dadurch gekennzeich net, daß die Polycarbonsäure und/oder deren Salze ein Molekulargewicht von 15 000 bis 28 000 aufweisen.
- Reinigungsmittel nach den Ansprüchen 1 oder 2, dadurch gekennzeichnet, daß die Polycarbonsäure und/oder deren Salze ein Molekulargewicht von 19 000 bis 22 000 aufweisen.
- Reinigungsmittel nach den Ansprüchen 1 bis 3, dadurch gekennzeichnet, daß sie solche Polycarbonsäuren und/oder deren Salze enthalten, bei denen zwischen zwei Carboxylgruppen 1 bis 3 Kohlenstoffatome stehen.
 - 5. Reinigungsmittel nach den Ansprüchen 1 bis 4, gekennzeichnet durch einen Gehalt an Polyacrylsäuren, Poly-

methacrylsäuren und/oder Polymerisationsprodukten von Maleinsäure und/oder deren Anhydrid und/oder Fumarsäure und Äthylen und/oder Propylen als Polycarbonsäuren.

- 5 6. Reinigungsmittel nach den Ansprüchen 1 bis 5, dadurch gekennzeichnet, daß die Polycarbonsäuren zumindest teilweise in Form ihrer Alkalimetall-, Ammonium- und/ oder substituierten Ammoniumsalze vorliegen.
- 7. Reinigungsmittel nach den Ansprüchen 1 bis 6, gekennzeichnet durch einen Gehalt von 1 bis 8 Gewichtsprozent
 Polycarbonsäuren und bzw. oder deren Salzen, bezogen
 auf das Gesamtgewicht des Reinigungsmittels.
- 15 8. Reinigungsmittel nach Anspruch 7, gekennzeichnet durch einen Gehalt von 2,5 bis 4 Gewichtsprozent Polycarbon-säuren und/oder deren Salzen, bezogen auf das Gesamt-gewicht des Reinigungsmittels.
- 20 9. Reinigungsmittel nach den Ansprüchen 1 bis 8, gekennzeichnet durch einen zusätzlichen Gehalt an phosphathaltigen Buildersubstanzen und/oder an chlorabspaltenden Verbindungen.
- Verfahren zur Herstellung von Reinigungsmitteln nach den Ansprüchen 1 bis 9, dadurch gekennzeichnet, daß man in einer Mischvorrichtung ein alkalibeständiges nicht-ionisches Tensid auf ein alkalisches Trägermaterial aufsprüht und unmittelbar nach dem Aufsprühen des Tensids eine Polycarbonsäure und/oder deren Salz mit einem Molekulargewicht von 12 000 bis 40 000 zugibt.

11. Verfahren zur Herstellung von Reinigungsmitteln nach den Ansprüchen 1 bis 9, dadurch gekennzeichnet, daß man in einer Mischvorrichtung ein alkalibeständiges Tensid mit einer Polycarbonsäure und/oder deren Salz mit einem Molekulargewicht von 12 000 bis 40 000 mischt und diesen Premix in einem zweiten Mischer einem alkalischen Trägermaterial zugibt.

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12. Verfahren zur Herstellung von Reinigungsmitteln nach den Ansprüchen 1 bis 9, dadurch gekennzeichnet, daß man einem Premix aus einem Trägermaterial, Polycarbonsäure und/oder deren Salz mit einem Molekulargewicht von 12 000 bis 40 000 und einem alkalibeständigen Tensid herstellt und diesen Premix in dem Hauptmischer auf die Hauptmenge des alkalischen Trägermaterials gibt.

EUROPÄISCHER RECHERCHENBERICHT

0132792 Nummer der Anmeldung

EP 84 10 8518

	EINSCHLA	GIGE DOKUMENTE		
Kategorie	Kennzeichnung des Dokum der mal	ents mit Angabe, soweit erforderlich, 3geblichen Telle	Betrifft Anspruch	KLASSIFIKATION DER ANMELDUNG (Int. Cl. ³)
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